INTERNATIONAL UNION OF CRYSTALLOGRAPHY

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STATEMENT

IUCr denounces the invasion of Ukraine



The International Union of Crystallography (IUCr) is greatly dismayed by the invasion of Ukraine and is concerned about its impact for the rest of the world. The IUCr stands in solidarity with the international scientific community as we are concerned for the well being and life of all Ukrainian people. We stand by their side. The IUCr seeks ways to serve all its members worldwide during this difficult time; it declares its support for the rights and freedoms conveyed in the Universal Declaration of Human Rights for all people.

The IUCr condemns all wars, and calls for a halt to hostilities and for a peaceful end to this crisis. We have suspended publication fees for our journals for authors based in the Ukraine (for more details contact support@iucr.org).

Hanna Dabkowska, IUCr President
Santiago García-Granda, IUCr Vice-President

LETTER FROM THE PRESIDENT

Let’s stay united



Hanna Dąbkowska

It is a difficult task to write a positive President’s Letter to you today. We are just stepping out from the long and difficult pandemic as we are watching in horror the development of the unjust and unprovoked but very deliberate war in Ukraine. Cities, lives and universities are being destroyed. Nobody expected it in the 21st century.

The IUCr denounces the war in Ukraine. We are greatly dismayed by the Russian military invasion of independent Ukraine, and we are concerned about its impact on the rest of the world. The IUCr stands in solidarity with the international scientific community as we are all concerned for the well-being and life of all Ukrainian people. Science must be seen as the stage set for dialogue and partnership between nations, even in times of geopolitical conflicts. The IUCr seeks ways to serve all its members worldwide during this difficult time; it declares its support for the rights and freedoms conveyed in the Universal Declaration of Human Rights for all people.

The IUCr condemns all wars, and calls for a halt to hostilities and for a peaceful end to this crisis.

Crystallography was always a science without borders, but now it is widening its scope to include more diversified topics as well as more diversified communities. We are about inclusion, not exclusion. When working in the IUCr we are all crystallographers and structural scientists. Not politicians, not social fighters, not tourists. Crystallography is a way of creative thinking. It is much more than just solving difficult structures or collecting and interpreting data. It reaches far beyond all of these; into creating new materials, modifying old ones, assessing resources on faraway planets, looking for new approaches to such ambitious targets as climate change, and promoting UN sustainability goals. The IUCr attitude is that we all have to collaborate to achieve these goals. We have to stay united and not allow the politicians to politicize our science. Let us hope that all scientists understand this.

We may not know what lies ahead for the world and for the IUCr, but at least we know what are we building upon.

The IUCr community has a lot to praise itself for. It is strong and vibrant, with more and more structural scientists joining our ranks. Financially – thanks to the IUCr Journals – we are doing reasonably well. When the world opens up – and settles down – we will be able to support young people willing to learn about crystallography at IUCr schools and meetings, and we will encourage our more experienced colleagues to enjoy sharing their knowledge, expertise and mentorship with the next generation of structural scientists.

To be able to do this, we need to stay consciously united even in this difficult time.

Many new initiatives have started recently and have proved to be very successful. The Rigaku School for Practical Crystallography ran again in January – thanks to Joe Ferrara and his team (see

https://japan.rigaku.com/sites/default/files/newsletters/times.2022.01.pdf). The school attracted over 500 students, with a significant proportion of them from India, US, UK, Germany and Poland.

The “Distinguished Lectures on Quantum Crystallography and Complementary Fields” organized virtually by Krzysztof Wozniak and Paulina Dominiak (from the IUCr Commission on Quantum Crystallography) are another welcome initiative. So far, the Lectures have gathered about 140 participants from all over the world (see https://qcrwebinar.chem.uw.edu.pl).

We are grateful to these organizers and to all the others for attracting more colleagues to the beauty and challenges of structural science, for encouraging them to read and publish in IUCr journals and to work on IUCr Commissions.

The COVID time was difficult; we missed many opportunities. No sabbaticals, no in-person meetings and courses, no travel to visit scientific friends and mentors. At the same time, we also learned new skills: how to effectively conduct Zoom conferences and meetings across the continents, perform experiments remotely in large facilities, or run a very successful hybrid Congress with more than 1500 participants.

These skills are already helping us. Preparations for the next IUCr Congress in Melbourne, Australia, in 2023 are well on the way. The International Program Committee (IPC), headed by Brendan Kennedy and Megan Maher, will be announced on the 26th IUCr Congress website soon. Commissions are working on the session proposals. Australia opened up recently for international travellers. Some IPC members can now attend the Program Meeting in person in Melbourne or in Prague, while others will join by Zoom. Many Commissions have delegated their Australian members to represent them in the IPC. First, at the time of nominations, travel to Australia was restricted. The second argument is that shorter travel reduces both the airfare costs and the carbon footprint related to long-haul flights. Being responsible citizens of the world, we need to take all of this into account.

On a different note: it is with mixed feelings that I am announcing that the IUCr Research & Development Officer and our long-time colleague, Brian McMahon, retired from his position at the end of 2021. We wish him all the best, and we hope that he stays in touch with the IUCr. His creative input into this organization during his 35 years of service cannot be overstated enough.

I am also signalling that Dr Alex Ashcroft has just recently decided to retire. Alex, who has served as our accomplished Executive Secretary since 2017, is still working with us and will be helping us to find his replacement. The advertisement for this position can be seen here.

As humans, we need each other to create a brighter future, for ourselves and for the next generations. Do not allow external forces to create divisions and ruin our strongest achievement: the 75-year-old IUCr community.

Let this approach be our conscious way of fighting for peace.

Please stay connected and stay safe.

EDITORIAL

Editorial



Optymistychna gypsum caves, near the village of Korolivka, Ukraine.

Mike Glazer

I never imagined that I would be writing an Editorial like this one day. The International Union of Crystallography was born in 1947 out of the ravages of the Second World War as a purely scientific union devoted to peace among scientists and the promotion and well-being of our wonderful science of crystallography and of crystallographers. For the last 75 years, the IUCr has served the worldwide community, treating all scientists as equals, irrespective of their nationality. The IUCr prides itself as a non-political society that encourages all scientists to treat each other equally. But, sadly, for the first time in its history, the IUCr has felt the need to issue an overtly political statement about the behaviour of a member country. Who would have thought it would come to this?

The Russian attack on Ukraine has come as a shock to us all. We all thought that those days were long past. When the Soviet Union fell, Russia became a much more open and modern society. Gone was that old repressive and grey country that I recall, as it began to embrace a more modern and normal life. There was a fantastic opportunity for it to become a normal member of the world democracy. But unfortunately, certain individuals were able to take advantage of the chaos of the time and capitalise on the way that many Russians have always felt threatened with being surrounded by perceived enemies. This allowed a leader straight out of the old KGB mentality to take over. What a lost opportunity for all of us!

Speaking personally, if I may, I have to emphasise that our Russian colleagues are not to blame for what has happened: they are victims of the system they have to live in. Over the years, I have visited Russia many times, the first time being for the IUCr Congress in 1966, during those cold war times. The experience of that first visit led me to my life-long support for the IUCr. I have met many Russians over the years and count lots of them as personal friends. I even had a friend during the old Soviet times who was obviously linked with the authorities: nonetheless, we used to meet regularly at IUCr Congresses and enjoyed getting together to have some drinks, tell stories and jokes, and argue about politics. Despite our different politics, we remained close friends. I recall many arguments about what was meant by freedom. He had his view, and I had mine. The last time I saw him was shortly before his death: he even came to Moscow, having taken the trouble to travel all the way from Chernobyl, where he was working on the after-effects of the reactor explosion, just to come and say hello to me when he learned that I was in Moscow.

I know that Russians are a warm and welcoming people, but unfortunately, they are held hostage by their own government. Since the invasion of Ukraine by Russian forces, I have not received any

communication from my friends there, and I am not sure if it is safe to contact them. I happen to have a personal connection and sympathy with four of the nations in the area (Poland, Belarus, Moldova and Ukraine), as all of my grandparents originally came from the region. My paternal grandmother came from Poltava in Ukraine. She was Russian speaking, and she was proud to have retained her original language. So, I feel particularly upset about what has transpired recently. We do not know how it will end, but let us hope that it will be soon and that we shall be able to again welcome our Russian colleagues in the true spirit of friendship.

By the way, we in the West often refer to these countries as being Eastern European and so somehow far from us. This needs a correction. They are in fact, Central European. It may come as a surprise to many of you to learn that the very centre of Europe is actually in the city of Polotsk, in … Belarus!



The geographical centre of Europe in Polotsk, Belarus (photo by Mike Glazer).

In this edition of the *IUCr Newsletter*, we have some new obituaries. One is for Ken Holmes, well known for his work in biological crystallography. Another is for someone who has been somewhat forgotten, June Lindsey (*née* Broomhead): her work around 1948 on purines played a vital role in the research into the structure of DNA. Also, Hans-Beat Bürgi has written about Jack Dunitz, who passed away last year.

Istvan Hargittai has written several interesting short articles for recent editions of the *IUCr Newsletter*. This time, he and Magdolna Hargittai have sent us one on quasicrystals. We know of Dan Shechtman's problems in trying to convince crystallographers that crystals of 5- and 10-fold symmetry were real. Perhaps less well appreciated is the work of Ágnes Csanády (*née* Bodoky), who was able to grow the world's most beautiful quasicrystals in the laboratory.

I have added an article myself about an interesting and unusual woman called Monica Maurice, sometimes known as "The Lady of the Lamp", whose connection with crystallography is somewhat mysterious. This goes back to a photograph taken in the early 1930s of the mineralogist and crystallographer Victor Mordechai Goldschmidt, which came into my possession when I moved from

Cambridge to Oxford in 1976. If any of you can add to the story, I would be pleased to hear from you.

We also have an article describing Crystallography in France today. You can read about the vast diversity of crystallographic research currently being carried out there. In addition, Thomas Proffen tells us about machine learning, John Helliwell considers whether the protein folding problem has now been solved, and Chris Sumby and Michael James write about the recent advances in the Australian Synchrotron. And then, you can read about the use of augmented and virtual reality at the Advanced Photon Source in Argonne.

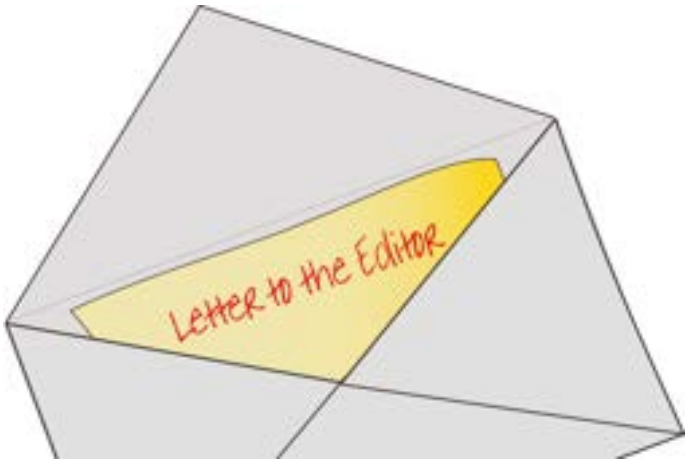
Also of interest, the Royal Society has now published a biographical memoir of Louise Johnson, who died in 2012. This is freely available to read here.

Finally, I should like to end this Editorial with a plea. As mentioned at the start, the IUCr was created 75 years ago to serve the worldwide community of crystallographers. Ten years later, it produced the first edition of the *World Directory of Crystallographers*, an unprecedented handbook of contact information and details of research interests tying together our community of structural scientists ever more closely. If you are not registered in the Directory or believe your details to be out of date, I would urge you to register here or update your entry here.

Shortly after writing this Editorial, I received from Polish colleagues the tragic news of the death of crystallographer Professor Vasyl Kladko of the V.E. Lashkaryov Institute of Semiconductor Physics, Ukraine, during a Russian attack in Ukraine. You can read more here.

LETTER TO THE EDITOR

Letters to the Editor



Reply to "Equality, equity, diversity, parity and discrimination"

I feel obliged to respond to Massimo Nespolo's letter, "Equality, equity, diversity, parity and discrimination" [*IUCr Newsletter* (2021). **29**(4)]. Nespolo wonders why, in an era of positive discrimination towards minorities, there is an "astonishing contrast with the deafening silence against other discriminations".

He expresses some concern as to whether women "are happy to be solicited because of their gender, rather than just because of their skills". I suggest that he discuss that concern with women.

A second issue of apparent concern rests on "discrimination" against the COVID-unvaccinated. In particular, he is worried about the upcoming Congress and General Assembly of 2023, to be held in Melbourne, "where the zero-COVID policy has enforced a severe deprivation of freedom and where freedom of speech is today brutally repressed when it comes to the “vaccines”, as live video recordings continue to show."

I am not associated with organisation of the Melbourne meeting, nor do I live in Melbourne. However, I am closer to the seat of "repression" than many other readers of Nespolo's letter, writing from Sydney. In short, his claims are nonsensical. There was violence on the streets and there was repression. In both cases, propagated by an odd people's army of conspiracy theorists, mirroring similar movements elsewhere, claiming to fight for the freedom of "the people" of Melbourne... the freedom to contract and spread COVID among a population who overwhelmingly and repeatedly agreed with a directive for certain workers to either get vaccinated or stay away from worksites.

Participation in 2023 in Melbourne will surely be afforded to "*bona fide* scientists", even if required to be fully vaccinated on entry to Melbourne. Objection to that requirement is anyone's right. However, that view contradicts a substantial corpus of *bona fide* science, so I see no grounds for concern.

Stephen Hyde, School of Chemistry, The University of Sydney, NSW, Australia
23 December 2021

Support for Ukraine: how scientific organizations and facilities perceive the military attack on Ukraine

The only “sin” committed by Ukraine was her wish to join the European Union and NATO, *i.e.* organizations offering economic and political stability and support in development to a country that had experienced the terror inflicted by the Red Army in 1918–1920, the Great Famine of 1932–1933, which took 10 million lives, and all the awfulnesses of the Second World War. The unprovoked and unjustified attack by Russia on Ukraine on 24 February 2022 started a war against civilians, patterned after the invasion of Poland in September 1939, when noncombatants were targeted and killed by the Nazi and Soviet military machines hellbent on breaking their spirit and willpower to defend their country against the invaders. The destruction of Kharkov, to name just one of many Ukrainian cities that suffered a similar fate, brings to mind a Saxon colonel Hoffmann artillery operation that led to a total destruction of Kalisz (a beautiful 2000-year-old city) and the air attack on little Wieluń (5000 victims after a single bombing). Both these Polish towns were systematically razed to the ground at the very beginning of WWI and WWII, respectively.

As I write these words, I am looking at a photograph of a destroyed apartment building (see below) in a residential district about 20 km from Kiev. It was hit by the aggressor tanks during the first days of the war, or should I say a "special military operation" (this official name suggests a peaceful action), which left one of the professors of Taras Shevchenko University (Kiev) homeless, as his flat was one of many that were hit (for the others, see the following photo). It is quite symbolic that a professor in solid-state physics is a target of a shell; he and his wife would not have survived if they had not noticed the first volley, and then had jumped straight to the underground shelter. This scientist and his younger colleagues were forced to leave their homes and they headed west, hoping to save a life. The bombing of towns and villages resulted in an unprecedented exodus of weeping women, children and older people at the scale of about 120,000 persons crossing the Ukrainian-Polish border each day (3.6 million in the month scale, counting this border, only).



Destroyed apartment of the scientist’s flat located near Kiev (middle segment, 2nd floor, left photo). Perforation of two neighboring buildings in the same settlement (right photo). The buildings were hit by shells fired from about 1.2 km distant tanks.

This brutal and devastating war has been condemned by all peace-loving societies which are now trying to help Ukraine in many different ways. In particular, multiple key scientific institutes and organizations took note of the unprecedented attacks and decided to voice their protest. A number of such protests were distributed even before the appeal of the Ukrainian scientists for strong support (dated 4 March 2022, see https://lnu.edu.ua/en/zvernennia-naukovtsiv-ta-osvitian-ukrainy-do-mizhnarodnoi-spilnoty/) signed by the Minister of Education and Sciences of Ukraine and many tens of Rectors and Directors. For example, the International Union of Crystallography denounces the invasion and calls for a halt to hostilities and for a peaceful end to this crisis (https://www.iucr.org/news/notices/announcements/iucr-denounces-the-invasion-of-ukraine). Protests of

Polish institutions can be found on their webpages, in particular that of the Polish Academy of Sciences (https://informacje.pan.pl/informacje/materialy-dla-prasy/3531-polska-akademia-nauk-solidarna-z-ukraina).

Two large scientific institutions, DESY (Hamburg) and MAX IV (Lund) operating powerful European synchrotron rings, widely distributed the information regarding the immediate cession of collaboration with scientists affiliated with the Russian Federation and its partner, Belarus; Belarus enables attacks on Kiev from sites close to its border with Ukraine. These institutions cancelled all the already allocated beamtimes with the participation of those scientists. Moreover, DESY and MAX IV strictly forbid publishing their scientific results jointly with researchers from Russian Federation or Belarus.

The Management of the ESRF, another world-leading synchrotron facility, "wishes to express its full solidarity with all colleagues and people affected, either directly or indirectly, by the ongoing tragic events following the aggression in Ukraine by the Russian Federation." New and stronger measures that express the ESRF protest against the Ukraine war are foreseen (https://www.esrf.fr/home/news/general/content-news/general/statement-following-the-aggression-in-ukraine-by-the-russian-federation.html). CERN (the operator of the LHC, the Large Hadron Collider) has suspended the observer status of the Russian Federation and stopped all collaboration. Multiple Swiss educational and scientific institutions strongly condemn Russia's military invasion and they "will do everything in their power to support and welcome their Ukrainian colleagues" (https://www.psi.ch/en/media/our-research/solidarity-with-ukraine); as we see, neutrality does not mean indifference.

The above decisions cut off the Russian Federation from modern research facilities serving for the most advanced investigations of matter. These decisions were taken by large facilities whose activities are chiefly based on international collaboration and so could be seen as damaging for their operations. However, as we understand it, the managements felt that it was their first duty to express their strongest condemnation of the aggression by the Russian Federation of Ukraine. And they used the means that were available to them, not only just words. The present bans do not target individual scientists, but are intended to demonstrate that the civilized world does not accept organizations that support the undoubtedly unjustified cruel act of aggression. Please show me any official scientific or other organization in the Russian Federation or Belarus that has protested against this war, if you disagree. No doubt, silence can be interpreted as tacit agreement.

Wojciech Paszkowicz, Institute of Physics, Polish Academy of Sciences, Warsaw, Poland
13 March 2022

We must not pretend that we do business as usual

The National Committee for Crystallography (NCC), which is the adhering body representing Poland in the IUCr, has proposed a motion to the IUCr Executive, to suspend the membership of Russia in the Union, with the motivation that the IUCr is an association of civilized members, and not of barbaric countries committing crimes on our brothers and sisters. Many learned societies and institutions throughout the world are undertaking all types of measures, to express their outrage at the barbarous acts committed by Russia in free and democratic Ukraine. A long list of official declarations can be found at the website of the International Science Council (https://council.science/current/news/statements-international-scientific-community-conflict-ukraine/), of which our Union is a member. Sadly, as of 13 March 2022, the IUCr is not listed there. These actions are not directed against our Russian colleagues, but are meant to condemn the lawless actions of Russia and to express our solidarity with Ukraine.

The vote within the NCC was overwhelmingly in favour of the petition. Two voices cautioned that such suspension may be counterproductive and may actually hurt Russian crystallographers, especially those in opposition to the official line, and – above all – arguing that science should be apolitical and should work to unite people and not to divide. But for such – quite rational – arguments, there are strong counter

arguments. Even if some pain will be inflicted on our Russian colleagues, the level of harm is beyond any comparison with the suffering of the innocent Ukrainians. And then, if we condemn, and indeed denounce, the inhuman and lawless behaviour of Russia, we can do this only with the weapons we have at our disposal. When innocent people are being killed by scores and displaced by millions, we cannot look the other way and pretend that business can be carried out as usual.

Mariusz Jaskólski and Marek Wołcyrz, on behalf of the Polish National Committee for Crystallography 13 March 2022 Editor's note: the IUCr declaration is now included on the International Science Council's website.

Over-reactions to the Ukraine conflict

The conflict in Ukraine has raised a wave of over-reactions, now spreading faster than SARS-CoV-2 in this Orwellian world where all those who do not comply with the allowed unique opinion are rapidly becoming outcast. Three examples:

The Cardiff Philharmonic has cancelled an all-Tchaikovsky programme
Perhaps they did not realize that Tchaikovsky died well before the birth of Putin?

Russian Paralympic athletes excluded from Beijing 2022
Handicapped athletes are clearly guilty of promoting this war!

The International Cat Federation bans Russian felines from competitions
As everybody knows, cats have huge responsibilities in this war!

I now see calls for banning Russian scientists from the forthcoming international congresses. Are those who propose such a ban aware of the basic policy of non-discrimination of the International Science Council (ISC), of which the IUCr is a member, according to which "International scientific meetings, events, teaching opportunities or research collaborations arranged or sponsored by the ISC itself or its members must be free from discrimination in attendance based on political or other opinion, ethnic origin, language, religion, citizenship, sex, gender identity, sexual orientation, disability, or age"?

I urge my fellow colleagues to avoid falling into the trap of Manicheism and guarantee the participation of Russian scientists, to show our solidarity with the victims of shameful discrimination striking people – even cats! – who are just "guilty" of being born in Russia, a country that has gifted the world with a huge number of treasures in art, literature and science.

Massimo Nespolo, Institut Jean Barriol, Faculté des Sciences et Technologies, Université de Lorraine, Vandoeuvre-lès-Nancy, France 13 March 2022

NOTICE

IUCr seeks an Executive Secretary (Chief Executive Officer)



The International Union of Crystallography (IUCr), which is both a global scientific union and a publisher of crystallographic journals and books, seeks an Executive Secretary (Chief Executive Officer) to manage the organisation and to administer its day-to-day operations.

The IUCr was founded 75 years ago to unite crystallographers worldwide and support them by providing a suite of bespoke research journals in which to publish their work, together with a set of specialised reference books. In addition, it has a long tradition of sponsoring schools, workshops and conferences globally through channelling financial support specifically to young scientists and furthering the development of crystallography in developing countries. All these activities are necessarily funded from the profits of the journals' publication business.

The IUCr has 53 adhering members – countries and regional committees – and it is incorporated in Switzerland as a "not-for-profit" organisation. It also runs 22 specialist commissions overseeing various aspects of crystallography. The Union is governed by a General Assembly of the adhering bodies that meets every three years. Between these meetings the responsibility for the work of the Union is delegated to the elected Executive Committee.

The IUCr is based at its headquarters in Chester, UK, in Abbey Square overlooking the Cathedral. Here it employs some 25 staff mainly engaged in the production of its 10 journals and the *International Tables for Crystallography*; seven of the journals are sold through the publisher Wiley and three are self-published. In addition there are teams of journal editors, co-editors and referees worldwide. The IUCr has an annual turnover in excess of USD 3 million and investments of a similar magnitude. The ability to handle financial matters, such as overseeing the preparation of the annual accounts by external accountants and dealing with taxation and investment matters, is required; attention to detail is a must.

The successful candidate is expected to direct the financial and administrative affairs of the Union and has support to help with accounting and financial matters. The Executive Secretary reports to the General Secretary and Treasurer on the Executive Committee, which is elected/re-elected every three years at the IUCr's congresses. The Union's Swiss registration means that meetings of the Executive Committee (annual) and the associated Finance Committee (twice a year) must take place outside the UK, and the Union's investments must also be held off-shore. The Executive Secretary must therefore be willing to travel overseas.

The IUCr offers a competitive salary in the range £60,000–£80,000 pa, with excellent benefits. To apply for the position please submit a CV, a covering letter detailing your suitability for the post, plus the names of at least two persons who can provide references, to Alex Ashcroft (execsec@iucr.org) by **20**

April 2022. In your submission please make reference to the summary below of the essential and desirable qualifications for this post.

The successful candidate must have the following:

An ability to manage people, possess excellent communication skills and have a basic knowledge of UK employment laws.

Excellent written and oral English skills.

Experience in the management, including the financial management, of projects.

An ability to work with scientists with diverse backgrounds from around the globe.

A willingness to travel outside the UK for IUCr committee meetings and major IUCr conferences and congresses.

Desirable attributes include the following:

A background in science, or in the organisation or promotion of science.

An interest in structural science and crystallography.

An interest in the scientific publications business.

Experience in an international organisation and planning activities.

Experience in a "not-for-profit" organisation.

Familiarity with financial investments.

NOTICE

Nominations invited for membership of IUCr Executive Committee and IUCr Commissions



IUCr Executive Committee 2021–2023.

In Melbourne in 2023, the 26th General Assembly of the IUCr will elect the new members of the Executive Committee and the Commissions. The Executive Committee wishes its own nominations to be representative of the desires of the community of crystallographers at large. We are therefore inviting the National Committees for Crystallography to submit suggestions for the future membership of the IUCr Executive Committee and the Commissions. It is important to have a fair representation of our diverse community, including young scientists and women, on our Executive Committee and Commissions, and ask that you take this into account when submitting your nominations. These suggestions will then be considered by the Executive Committee when preparing its own nominations at its meeting in 2022.

Executive Committee

The vacancies to be filled are those of President, Vice-President, General Secretary and Treasurer and three ordinary members. Of the offices to be filled, only the General Secretary and Treasurer is eligible for re-election to the same office. We invite you to make suggestions for each vacant office. Please feel free to suggest more than one person for any particular position. Although a National Committee has special knowledge of crystallographers in its own country, and we would be pleased to have suggestions based on this knowledge, it would also be helpful if you suggested candidates from other countries as well. The Executive Committee should be a fair representation of the variety of nationalities from the different continents that make up the membership of the IUCr and of the variety of fields and sub-disciplines in our science. A good gender balance is also important. The nomination form is available here; the submission deadline is **30 June 2022**.

Non-publishing Commissions

The Executive Committee welcomes suggestions from National Committees for the Chairs and members of the non-publishing Commissions. The current memberships can be seen here. Your suggestions will be of great help to the Commissions when they compile their own recommendations, which form the basis from which the Executive Committee's nominations to the General Assembly for the Chairs and members of Commissions are compiled. The IUCr needs candidates who are willing to participate actively in the work of the Commissions, and it would be most helpful if you could include some brief information on the specific scientific interests of the persons you suggest. The nomination form is available here; the submission deadline is **30 June 2022**.

IUCR CONGRESS

IUCr 2023 workshop applications now open



Express your interest to run a workshop at IUCr 2023

The Local Organising Committee of IUCr 2023 is excited to announce that they are now accepting expressions of interest for individuals or groups to hold workshops immediately prior to the Congress.

Workshops will be held on Monday 21 and Tuesday 22 August 2023.

Workshops can be half-day, full day or two full days in length, and should be unique content that would fall outside the usual meeting and symposium format occurring during the Congress itself.

If you would like to host a workshop for IUCr 2023 please see the full requirements and terms and conditions on the website and complete the Expression of Interest form **by midnight (AEST) on Thursday 21 July 2022**.

Other key dates

Call to action

Date

Call for abstracts open

23 August 2022

Bursary applications open

23 August 2022

Call for abstracts close

21 November 2022

Bursary applications close

21 November 2022

Notification of abstract acceptance

1 February 2023

Early bird registration deadline

15 February 2023

Deadline for presenters to register

15 February 2023

Accommodation booking deadline

22 July 2023

IUCr 2023 pre-Congress workshops

21 & 22 August 2023

IUCr 2023 Congress

23–29 August 2023

To sign up for IUCr 2023 notifications, please go here. IUCr 2023 posters for printing or electronic circulation to your colleagues are available here. Thank you!



IUCR ACTIVITIES

Call for nominations for the thirteenth Ewald Prize



The IUCr invites your nominations for the Ewald Prize for outstanding contributions to the science of crystallography. The Prize is named after Paul P. Ewald, in recognition of his significant contributions to the foundations of crystallography and to the founding of the IUCr. Professor Ewald was the President of the Provisional International Crystallographic Committee from 1946 to 1948, the first editor of the IUCr journal *Acta Crystallographica* from 1948 to 1959 and the President of the IUCr from 1960 to 1963.

The Prize is presented once every three years during the triennial International Congresses of Crystallography and consists of a medal, a certificate and a financial award. The recipients to date are as follows:

Year

Place

Recipient(s)

1987

Perth, Australia

Professor J. M. Cowley and Dr A. F. Moodie

1990

Bordeaux, France

Professor B. K. Vainshtein

1993

Beijing, China

Professor N. Kato

1996

Seattle, WA, USA

Professor M. G. Rossmann

1999

Glasgow, UK

Professor G. N. Ramachandran

2002

Geneva, Switzerland

Professor M. M. Woolfson

2005

Florence, Italy

Professor P. Coppens

2008

Osaka, Japan

Dr D. Sayre

2011

Madrid, Spain

Professor E. Dodson, Professor C. Giacovazzo and Professor G. M. Sheldrick

2014

Montreal, Canada

Professor A. Janner and Professor T. W. J. M. Janssen

2017

Hyderabad, India

Professor T. L. Blundell

2020

Prague, Czech Republic

Dr Olga Kennard, OBE FRS

The thirteenth Prize, for which nominations are now being invited, will be presented at the Melbourne Congress in August 2023.

Scientists who have made contributions of exceptional distinction to the science of crystallography are eligible for the Prize, irrespective of gender, nationality, age or experience. The Selection Committee will give careful attention to the nominations of outstanding scientists who have not yet won a Nobel Prize. Either an exceptionally distinguished scientific career or a major scientific accomplishment may be recognised. Current members of the Selection Committee and the President of the IUCr are not eligible. No restrictions are placed on the time or the means of publication of the nominee's contributions. The prize may be shared by more than one contributor, but no more than three, to the same scientific achievement.

Nominations for the thirteenth Ewald Prize should be submitted electronically using the Ewald Prize Nomination Form to the IUCr Executive Secretary at execsec@iucr.org. The closing date for nominations is **31 August 2022.**

IUCR ACTIVITIES

Call for nominations for the second W. H. & W. L. Bragg Prize



James Fraser (left) and Jean-Philippe Julien, winners of the inaugural W. H. and W. L. Bragg Prize for outstanding early-career crystallographers.

The IUCr is pleased to invite nominations for the second W. H. & W. L. Bragg Prize for outstanding contributions to the science of crystallography. The Prize is named after Professors W. H. and W. L. Bragg, in recognition of their significant contributions to the foundations of crystallography. Professors William Henry and William Lawrence Bragg pioneered the use of X-rays as an instrument for the analysis of crystal structure, and were the joint recipients of the 1915 Nobel Prize for Physics.

The Prize consists of a certificate and a financial award, and is presented once every three years during the triennial International Congresses of Crystallography. The inaugural Prize was awarded in Prague, Czech Republic, in 2020 to Professor James Fraser (USA) and Dr Jean-Philippe Julien (Canada). The second edition of this Prize, for which nominations are now being invited, will be presented at the Melbourne Congress in August 2023.

Unlike the Ewald Prize, the W. H. & W. L. Bragg Prize is awarded to a scientist in the early stages of his or her career, irrespective of gender, nationality or age. Nominees will be expected to have registered in the World Directory of Crystallographers.

Nominations for the second W. H. & W. L. Bragg Prize should be submitted electronically using the Bragg Prize Nomination Form to the IUCr Executive Secretary at execsec@iucr.org, from whom copies of the names of the Selection Committee may be obtained. Supporters and the nominee will then be provided with forms to complete. The closing date for nominations is **31 August 2022**.

OUTREACH

IUCr collaborates with physics students worldwide



IAPS School Day 2019 in Noida, India.

The IUCr and the International Association of Physics Students (IAPS) have formalised their collaboration by signing a Memorandum of Understanding (MoU), which was accepted by IAPS members in August 2021.

IAPS is a non-profit non-governmental organisation run entirely for and by physics students from around the world, with over 65,000 members. Formed in 1987 in Hungary and now based in the European Physical Society (EPS) headquarters in France, IAPS is present in all the continents of the world, with its members being National or Local Committees or individual students.

The idea of an association like this was formed in the first edition of the International Conference of Physics Students (ICPS) in 1986. Nowadays, ICPS is the major event of IAPS, where roughly 400 students from 50 countries meet annually to discuss physics, present their projects and socialise. Moreover, it fosters a physics competition for teams of bachelor and master students, namely PLANCKS, which also takes place every year. These events are organised by the IAPS members, who must bid at the IAPS Annual General Meeting. IAPS also organises and promotes several scientific excursions and conferences, such as iaps2CERN.



Participants at the International Conference of Physics Students 2017 in Turin, Italy (© ICPS 2017).

Besides scientific events, IAPS is also a supporter of outreach and advocacy initiatives. With the IAPS School Day event, hosted yearly around the World Science Day for Peace and Development on 10 November, it supports and incentivises its member committees to visit schools and showcase several physics experiments. Furthermore, it offers support and funding to their members to organise a variety of outreach events throughout the year. It is now a high-level Endorsing Partner of the Steering Committee of the International Day of Light, having created the IAPS Light week in 2018, and is an Affiliated Commission of the International Union of Pure and Applied Physics (IUPAP). IAPS has worked continuously to support friendly relations and collaboration between physics students, also supporting its members in their academic and professional work, as well as discussing and acting on scientific, social and cultural issues.

Through the MoU between IAPS and IUCr, the IAPS members will discover the IUCr, its work and the field of crystallography and its importance, with participation in the IUCr events being promoted to the physics student community. Collaborations will also be fostered to organise new activities or support existing ones, especially outreach initiatives. As such, this MoU is the first step in the promising cooperation between IUCr and IAPS.

Contacts

Ruhi Chitre, President of the IAPS Executive Committee, president@iaps.info; Sofia Ferreira Teixeira, IAPS–IUCr Liaison, sofia.ferreira.teixeira@iaps.info.

For more information regarding IAPS, its members and activities and other partners, please visit https://www.iaps.info/.

IUCR JOURNALS NEWS

Modern approaches and tools for teaching crystallography – call for papers for special issue

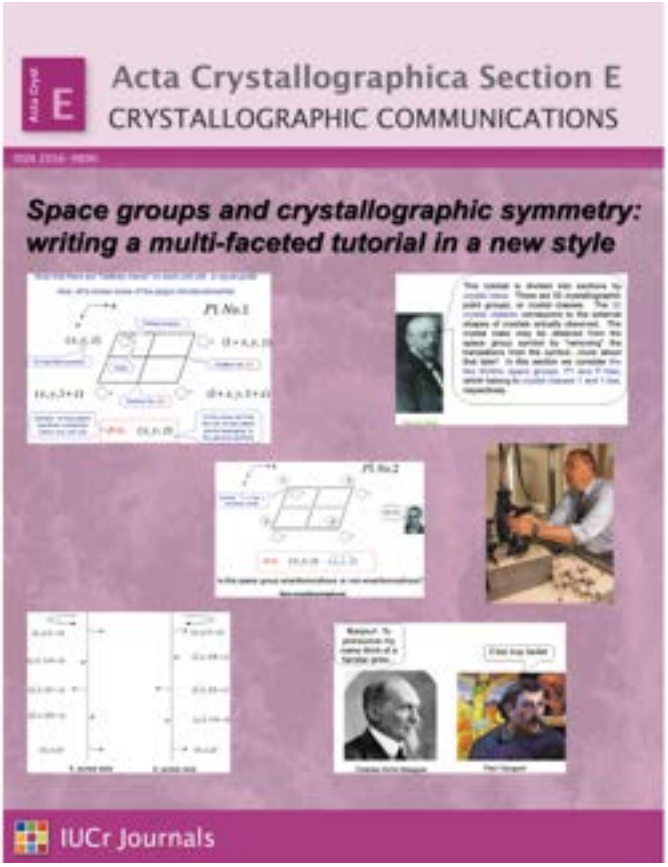


Graciela Díaz de Delgado, Sean Parkin

Acta Crystallographica Section E: Crystallographic Communications is the journal of choice for the publication of high-quality structure determinations of compounds of a very diverse nature. Many of these studies involve the use of advanced concepts to solve difficult problems such as twinning, disorder, modulated structures *etc*. Reports on the determination of challenging structures can provide a good opportunity to show young crystallographers and newcomers to crystallography how to approach these difficult problems. The global health crisis caused by the COVID-19 pandemic has had a negative impact in our field as many laboratories have had to close for long periods of time. However, scientists have continued working remotely, teaching online courses and tutoring students using different electronic meeting platforms.

Following the long teaching tradition of the IUCr, we would like to encourage you to submit manuscripts to *Acta Cryst. E* that can be used as educational materials so that the younger generation may benefit from the knowledge and expertise of experienced crystallographers.

In this new environment, we would particularly like to receive articles based on experiences of teaching crystallography online and of how best to use some of the facilities available to access diffraction equipment remotely. Articles giving practical advice on using crystallographic software to address different aspects of structure determination, refinement, graphics, analysis *etc*. would also be welcome. Besides the main text of the manuscript, we encourage the submission of supporting information containing comprehensive practical examples, instructions, software input files, reflection data and results. Contributions on new tools for teaching the fundamentals of crystallography would also be of interest. Examples of articles with a strong teaching component that have been published recently in our journal are available here.



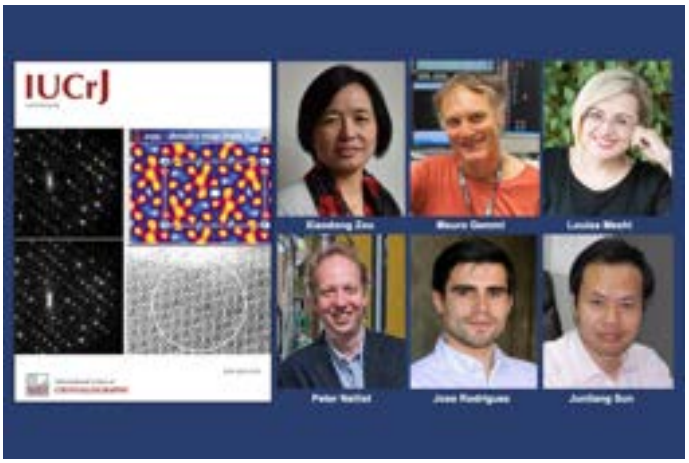
All manuscripts for this special issue should be submitted by 30 June 2022. They will be subject to the normal peer-review process.

We look forward to your submissions!

Acta Cryst. E is an open-access journal. For more information, please go here.

IUCR JOURNALS NEWS

IUCrJ launches electron crystallography section



Recent years have seen the rapid development and expansion of electron crystallography, much of it highlighted at the IUCr Congress in Prague last year. This has made it increasingly important to establish a home within IUCr journals for high-quality, high-impact papers in this field. The International Union of Crystallography is therefore pleased to announce the launch of a new section of **IUCrJ** dedicated to electron crystallography.

As announced in the January issue of the journal, Professor Xiaodong Zou (Stockholm University, Sweden) has been appointed as Main Editor of the electron crystallography section. Five new Co-editors will work with Professor Zou – Mauro Gemmi, Louisa Meshi, Peter Nellist, Jose Rodriguez and Junliang Sun – and will aim to attract articles reporting important electron crystallography results and breakthroughs to the journals. **IUCrJ** welcomes these new Co-editors and below we provide brief biographical details.

Mauro Gemmi (Istituto Italiano di Tecnologia, Pontedera, Italy) has worked in several electron microscopy labs in Europe (Stockholm University, Milan University, Institut Ne´el Grenoble) becoming an expert in the application of electron diffraction to structure solution problems. Since 2015 he has been the PI responsible for the Electron Crystallography research line of the Istituto Italiano di Tecnologia. He has been among the first to extensively use precession electron diffraction for solving crystal structures. His main research goal has been to apply electron diffraction to structural problems in any field of crystallography. His scientific dream is to see electron diffractometers in every crystallographic lab.

Louisa Meshi (Ben-Gurion University of the Negev, Beer-Sheva, Israel) specializes in structure solution of complex intermetallics applying precession electron diffraction (PED), convergent beam electron diffraction (CBED) and 3D electron diffraction tomography methods. In her laboratory, structure solution of these phases (appearing as nano-sized precipitates in metallic matrices) is combined with characterization of line and planar crystalline defects (using various electron imaging and diffraction methods), aiming at development of structural materials of tomorrow.

Peter Nellist FRS (University of Oxford, United Kingdom). His research centres on the applications and development of high-resolution electron microscope techniques, in particular scanning transmission electron microscopy (STEM) including 4D STEM diffraction-imaging techniques, *Z*-contrast imaging, electron energy-loss spectroscopy and applications of spherical aberration correctors. His technique development work includes ptychography, quantification of diffraction and image data, methods for the three-dimensional imaging and spectroscopy of materials using optical sectioning, and methods to allow high-resolution imaging and spectroscopy of radiation sensitive materials. Applications include Li-ion battery materials, hybrid organic–inorganic perovskites, polymer crystallinity and catalysts.

Jose Rodriguez (UCLA, Los Angeles, USA) studies the complex architecture of biological systems – from single biomolecules to cellular assemblies – at high resolution. His work is largely based on diffraction phenomena and combines computational, biochemical and biophysical experiments. The development of new methods is central to this work, particularly using emerging technologies in cryo-electron microscopy, nano and coherent X-ray diffraction, and macromolecular design. Combined, these tools can reveal undiscovered structures that broadly influence chemistry, biology and medicine.

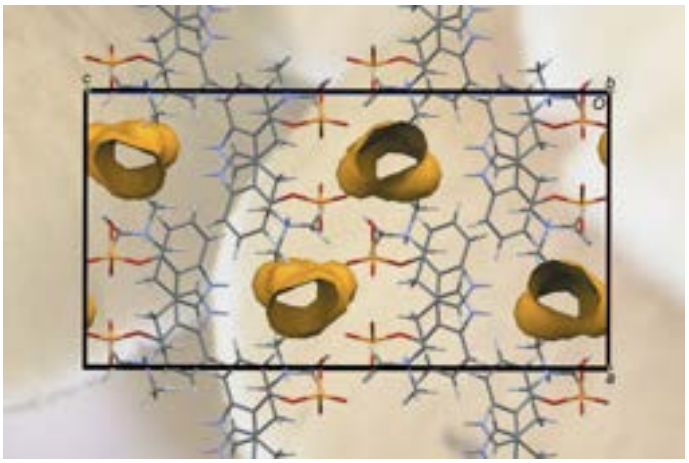
Junliang Sun (Peking University, Beijing, China) works mainly on the development of structure characterization methods (electron crystallography and powder X-ray diffraction) and the synthesis of inorganic materials. Among the 245 approved zeolite topologies, he has been involved the discovery of ten topologies. In relation to structure characterization methods, Junliang and his co-workers have developed rotation electron diffraction which pushed the size limitation for single-crystal diffraction to tens of nanometres. Combined with powder X-ray diffraction, this has allowed series of difficult structure problems to be solved.

More information about the Editors, together with a selection of electron crystallography papers already published in the journal can be found at https://journals.iucr.org/m/services/editors_electron_cryst.html. We hope **IUCrJ** will become the natural home for your innovative research in electron crystallography and look forward to receiving your articles.

This article was originally published in *IUCrJ* (2022). **9**, 163–164.

COMMENTARY

Crystallographic hallucinations



Brian H. Toby

Psilocybin was of great interest to some of my contemporaries during my student days, but not due to its crystal structure, at least not directly. At present, due to the study of hallucinogens as possible treatment agents for various mental illnesses, their use has become a topic of significantly renewed interest (Gill *et al*., 2020; Sherwood & Prisinzano, 2018). As is well known in the pharmaceutical industry, crystallographic characterization of materials is crucial in order to understand the bioavailability of a drug (Byrn *et al*., 1999). Structural knowledge is thus highly important for the potential use of psilocybin.

In their detailed study of the structural forms of psilocybin reported recently in *Acta Crystallographica Section C*, Sherwood *et al.* (2022) solve two novel anhydrous psilocybin structures *ab initio* from high-resolution synchrotron and from laboratory X-ray powder data, respectively. While attempts to directly crystallize the anhydrous forms were attempted, they could only produce the phases by removing water from a hydrated form. The hydrated phase forms single crystals and has a determined structure, but dehydration destroys these crystals, producing crystalline powders. To confirm the quality of the powder structures and review the hydrogen-bonding motifs, they performed computations with plane-wave density functional theory (DFT). A second form of DFT was used in ‘gas phase’ computations to compare the energetics of the psilocybin conformations for isolated molecules for their two new polymorphs and the previously-known hydrated form.

While the very high quality of the fits between the observed and calculated patterns would already give one confidence in their results, their verification that that calculations from theory demonstrate that the structures represent energetic minima boosts confidence in their results manyfold. However, it had been reported in the patent literature that different polymorphs exist. To address this, Sherwood *et al.* (2022) performed Rietveld fits on data from a total of 24 different samples, either with contemporary data collection or with digitized powder diffraction patterns taken from the literature. They show that all these patterns are consistent with their two new anhydrous structures and the known hydrous form, sometimes as mixtures and sometimes where intensities require correction for significant preferred orientation. This texture is not at all surprising considering the highly anisotropic crystal habits they see in the powders. Furthermore, they made significant efforts to coax psilocybin to crystallize in other structural forms, but to no avail. Based on all this, the previous assignment of other phases for anhydrous psilocybin seems highly unlikely and this careful work casts doubt that any other anhydrous polymorphs could exist.

This *tour de force* study is one that deserves exposure in the classroom, so that crystallography students may see how powder diffraction crystallography can rise to the gold standard level that we expect from single-crystal analysis. In fact, it is worth noting that the agreement seen between DFT and the powder–synchrotron structure is far better than the DFT–single crystal agreement. The impact of this work on patents issued with incorrect crystallography is something to be resolved in courts, not the

scientific literature, but I have no doubt that a number of scientific managers are currently considering the potential impact that less-than-careful crystallography might have on their organization's intellectual property collections.

The compelling structural validation work by Sherwood *et al*. (2022) demonstrates the great value of DFT as a partner tool to crystallography. As I write this, on the eve of International Holocaust Remembrance Day, thinking about DFT raises for me thoughts on another topic of contemporary worldwide interest – if not contention – which is on the value in opening borders for immigration. It is worth remembering that the life of Walter Kohn, who won the Nobel Prize for his development of DFT, was saved by the British Kindertransport, which resettled children in the UK even when their parents could not cross borders. As a child, Walter and his sister Minna were able to leave Austria for Britain and thus survived World War II. Walter was educated in Canada and eventually immigrated to the United States. Walter's work gave the world this highly useful approach to quantum theory, but he never saw his parents again; they died in Auschwitz (Kohn, 1998; https:// www.notablebiographies.com/supp/Supplement-Ka-M/KohnWalter.html).

As a final point, with the ubiquitous availability of fast computers and with easier to learn codes (some open source), DFT is an increasingly accessible tool for crystallographers. Even with a good-quality single-crystal structure, verification of hydrogen-bonding networks is a worthwhile exercise. With powder diffraction crystallography, structural validation becomes even more valuable.

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COMMENTARY

Negative linear compressibility at extreme pressure

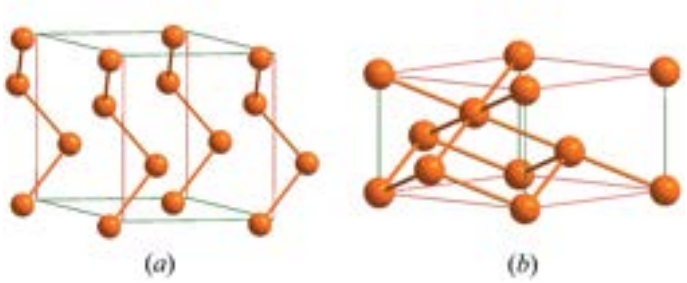


Figure 1. The crystal structures of Se-I (*a*) and Se-V′ (*b*). Unit-cell edges in the directions that contract with increasing pressure are shown in green, while those in the directions that expand with increasing pressure are shown in red.

Kamil F. Dziubek

Although the term allotropy, which signifies the ability of chemical elements to exist in different structural modifications, was introduced as early as 1840 by Jöns Jakob Berzelius (Berzelius 1841; Jensen, 2006), the number of known elements that existed in more than one polymorph was quite limited at this time. It was only later that studies under various pressure and temperature conditions became possible with the development of high-pressure experimental techniques, sparking a rash of newly discovered crystal forms, often exhibiting unusual structures and properties (Young, 1991; Tonkov & Ponyatovsky, 2005).

While many elements have been thoroughly studied under non-ambient conditions, there are still many controversies regarding their phase diagrams. In particular, phase boundaries in the pressure–temperature diagram, the sequence of phase transitions and the structure of particular phases often become the subject of debate. Inconsistencies and contradictory results concern mainly extreme pressures, where the minute size of the sample, substantial nonhydrostatic stress and other factors, like kinetic effects, may significantly affect the results. With the advent of third- and fourth-generation synchrotrons, the X-ray beam is focused down to micron or even submicron size enhancing spatial resolution. In addition, the development of detectors with improved signal-to-noise ratio and fast readout has further enhanced the capabilities of the instruments. For this reason, numerous early studies have been challenged and revised recently.

Systematic investigation of group 16 elements is fascinating on many levels. While the lighter elements – oxygen and sulfur – form molecular crystals, and polonium, the heaviest element of the group observed in nature, is metallic, both selenium and tellurium are semiconductors. As a consequence, elemental selenium found numerous applications ranging from photocopying (Chou *et al*., 2003) to flat-panel X-ray detectors (Huang & Abbaszadeh, 2020). Understanding the behaviour of compressed selenium is therefore of great interest for both fundamental physics and industrial applications. Below 60 GPa selenium exists in four various crystal phases. According to Akahama *et al*. (2021), the rhombohedral phase named Se-V (space group *R*3*m*, β-Po structure) occurs between ~60 GPa and 140 GPa, where it transforms to the Se-VI b.c.c. phase, stable up to at least 317 GPa.

In the March 2022 issue of **IUCrJ**, Yuan *et al.* (2022) revised the extreme pressure phase transitions in selenium and reported a hitherto unknown phase, characterized by an anomalous linear expansion in one principal direction under hydrostatic pressure. This finding was possible only thanks to the state-of-the-art data collection technique. The sample was subjected to constant compression and short-time exposure using a fast detector. In this way, the authors collected more than 4300 diffraction images from ambient pressure to 210 GPa, in fine pressure steps. Careful analysis of the results revealed that the variation of Se-V lattice parameter *a* with pressure changes the slope from decreasing to increasing at 120 GPa. This trend is maintained until 148 GPa, and the Bragg peaks of the rhombohedral phase are visible up to 160 GPa.

Negative linear compressibility (NLC) is a rare phenomenon where a crystal expands along one direction under hydrostatic compression (Cairns & Goodwin, 2015). It has been revealed in many classes of materials, ranging from inorganic oxides and salts to metal-organic frameworks and molecular crystals. Two of the very few examples of NLC among chemical elements are the trigonal ambient-condition polymorphs of selenium and tellurium (space group $P3_121$), Se-I and Te-I (Keller *et al.*, 1977). In these isostructural phases, atoms are covalently bonded in helical structures, which interact with one another through weak intermolecular forces. External pressure increase reduces interhelical distances while slightly increasing the intrinsic pitch of helices. In contrast, in Se-V each selenium atom is involved in a three-dimensional covalently bonded framework with six nearest neighbours within bonding distance (Fig. 1). This Se–Se bond is much less compressible than the second-nearest neighbour contacts, which in consequence leads to structural rearrangements and the expansion of lattice parameter *a* with pressure above 120 GPa. Cairns & Goodwin (2015) attributed such a microscopic mechanism of NLC to correlated polyhedral tilts. Noteworthy, the absolute value of NLC in Se-V is around one order smaller than that of Se-I. Hence, in one simple material, two different phases exhibit NLC in various pressure range, and in each case the mechanism can be rationalized differently at the atomic level.

Yuan *et al*. (2022) reported the new phase of elemental selenium, isostructural to Se-V, but with different properties demonstrated by its NLC. To distinguish these structures they suggested the name Se-V' for rhombohedral selenium above 120 GPa, the pressure at which the parameter *a* begins to increase. It should be emphasized, however, that this notable discovery of NLC above one megabar could not have been accomplished without modern synchrotron radiation facilities and cutting-edge experimental techniques. Constantly evolving innovations and solutions stimulate high-pressure researchers to come up with even more daring ideas. After all, matter under extreme conditions still has many secrets to be discovered.

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COMMENTARY

Sifting fluctuation scattering from microtextured samples

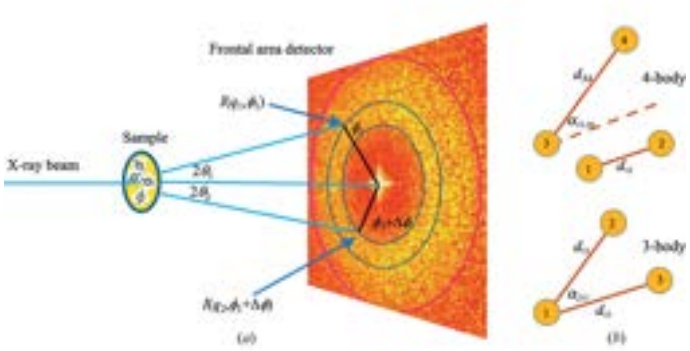


Figure 1. (*a*) The power of FXS is that, summing the products of intensities with two different q values and with constant angular separation $\Delta\phi$, one obtains not only the interatomic vector distance lengths as from standard 1D PDF, but also (*b*) the angle between two interatomic vectors. These can share a common atom (three-body correlation) or not (four-body correlation).

Antonio Cervellino

The paper by Binns and coworkers in the March 2022 issue of **IUCrJ** (Binns *et al*., 2022) contains a key finding for promoting the structural investigation of matter exhibiting complex order by fluctuation X-ray scattering methods (FXS). As a result of the deep and accurate theoretical analysis thereby exposed, substantiated by careful experiments, one of the strongest limits of the technique, its sensitivity to microstructure, has been overcome.

A brief outline is in order. It is nowadays assumed that by X-ray diffraction we can determine the atomic structure of a crystal. Much of the structural information is destroyed when we have a large number of isotropically and homogeneously oriented crystals instead of a single one (as in a powder). The directional averaging means that the diffraction pattern is substantially 1D. If sharp Bragg peaks are the dominant feature of the pattern, they can be lifted in 3D momentum space and the structure can be solved as well, albeit with more difficulty and some limitations. For a powder with particles that are not (or are imperfectly) crystalline, we can reliably recover only the scalar pair correlation or pair distribution function (PDF) from the scattered intensity, but no higher correlations. So a complex deduction game must be played to translate interatomic distances – directly gleamed as sharp peaks of the PDF – into 3D geometry. Kam (1977) thought to exploit the then novel 2D detectors and add fluctuation X-ray scattering (FXS) into the frame. FXS is a minor component of the scattering from an ideally isotropic powder, arising from zero-average fluctuations of the orientation distribution of the constituent particles. Separating FXS after subtracting the powder signal (the constant-momentum average of the pattern) was shown to yield higher-level information, namely the pair-angle distribution function (PADF) (Martin, 2017), that includes three- and four-body correlations with distances and angles (*cf*. Fig. 1).

Clearly, the exploitation of FXS to yield PADF brings us much closer to understanding structures of less-crystalline matter (*e.g.* liquid crystals and nanoparticles within or without self-organized supercrystals). The FXS signal is, however, weak and can only be extracted if we can subtract the baseline powder diffraction signal. This is straightforward with ideal powders.

Enter preferred orientation (texture) and the situation becomes much more complex. Basically, texture introduces an *a priori* unknown angular variation of the baseline powder signal, which is then added to the FXS signal but does not contain structural information, while easily strong enough to swamp the FXS signal. Texture has long since been the bane of powder crystallography; its effects can be dramatic in falsifying the Bragg intensities, and it also affects the PDF (Cervellino & Frison, 2020). Huge efforts have been devoted to understanding and characterizing texture (Roe, 1965; Bunge, 1987) in order to develop useful correction algorithms. Moreover, sample preparation methods have been developed to reduce *macro*texture – the texture that is determined mainly by sample preparation and containment, and whose description is unique for the whole sample. However, there is often also *micro*texture – preferred orientation on a local scale, much bigger than the powder particles but much finer than the sample dimension. This is locally variable, depending on local conditions, so no sample-wide correction is possible. It is also typically not so strong as macrotexture can be; however, it can still easily be stronger than FXS. Not always of course, otherwise we would not have had a successful history of FXS experiments. Microtexture adds this margin of unpredictability that strongly dampens expectations of success in an experimental campaign based on FXS.

The importance of Binns and coworkers' paper (Binns *et al*., 2022) is that it provides an accurate analysis of the effects of microtexture on FXS. It also presents an important result in terms of the intensity ratio of the microtexture signal to the FXS one. This ratio scales linearly with the number of illuminated grains, *i.e.* with the illuminated sample volume. This means that thinner beams will give more relevance to the FXS signal. This is especially good news because it leverages the great developments of the last decade (Shin, 2021) at synchrotron and XFEL sources (but also in laboratory instruments) in terms of brilliance. Between many other positive effects, a high brilliance facilitates the production of stable microfocused beams, down to well below the micrometre range. Increasing source brilliance means that very thin beams are still powerful enough to give a clear FXS signal within the necessarily short acquisition time while depressing the microtexture signal into irrelevance by their tiny size. In Binns *et al.* (2022) a brilliant experimental test is shown to highlight this feature. An experiment with a large beam (a few thousands of square micrometres) shows only the microtexture signal, while another experiment with a thin beam (a couple of square micrometres) on the same substance (a well reproducible liquid crystal mesophase) clearly showed the FXS signal.

And this is just the beginning, of course. A clear linear scaling behaviour of the FXS to texture ratio will open the door to more complex but still easily feasible experimental setups. These may involve rapidly variable beam spot size coupled with fast area detectors and principal component analysis. And FXS could be poised to become a mainstream method, finally leaving its obscure niche.

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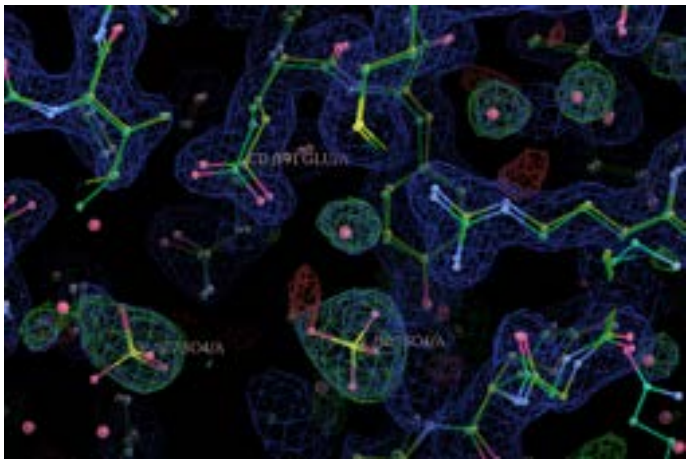
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FEATURE ARTICLE

The protein folding problem is solved. But the debate continues



John R. Helliwell

I have had two earlier articles in the *IUCr Newsletter* on this important research topic, the protein folding problem, including its long history (Helliwell, 2020, 2021). Firstly, I described the "everyone agreed breakthrough," especially endorsed by the judges at the Critical Assessment of Techniques for Protein Structure Prediction's 14th iteration "CASP14" of the DeepMind's entry, *AlphaFold*. Secondly, I described the sudden, indeed breathtaking, announcement (Cusack *et al*., 2021) of the collaboration of DeepMind with the EMBL EBI (European Molecular Biology European Bioinformatics Institute), whereby hundreds of thousands of predicted three-dimensional structures across 20 species were made available to the structural biology community. This gave me the chance to evaluate for myself, with one of my favourite protein crystal structures, the enzyme from sheep liver 6-phosphogluconate dehydrogenase, just how well *AlphaFold*2 performed. The sheep enzyme was not included in the species of the 20 provided, so instead I downloaded the three-dimensional structure for the human 6-phosphogluconate dehydrogenase amino acid sequence. It is a favourite because it was the subject of my DPhil project at the University of Oxford from 1974 to 1977 (Adams, Helliwell & Bugg, 1977). I refined the *AlphaFold*-predicted structure against the 2.0 Å X-ray diffraction data 2pgd (Phillips, Gover & Adams, 1995); the X-ray study had moved on from the 6 Å and 2.6 Å studies of my thesis. The difference electron-density map clearly showed a sulfate ion, which I showed as evidence to the readers of my article.

Not long after my article came out, I received the University of Washington *Robetta* fold prediction (https://robetta.bakerlab.org/; Baek *et al.*, 2021) for the sheep liver 6-phosphogluconate dehydrogenase amino acid sequence that I had submitted. This did not have as good model refinement statistics or electron-density fit, but the changes needed to the predicted structure were clear from the difference map. I duly contacted Mike Glazer, as Editor, on whether an update should be provided, but we decided that the story was already clear enough. The prediction method worked, or, I can say now, both methods, *i.e.* DeepMind and *Robetta*, worked. I concluded, on this evidence, that I could have saved about a year of my DPhil time spent on solving the protein crystallographic phase problem by using these predicted structures. There were caveats though:

They were predictions, which Tom Terwilliger, a former Chair of the IUCr Commission on Biological Macromolecules, described in a recent virtual lecture entitled "*AlphaFold changes everything: incorporating predicted models in X-ray and Cryo-EM structure determination*" hosted by the University of Strasbourg, as "*hypotheses*".

It was oft-repeated that these were accurate structures, sometimes by DeepMind itself, but I hung onto their correct way of describing their structures as needing rigorous assessment via experimental data. To

their credit, they had also assigned a confidence estimate to each portion of their predicted protein structure.

I emphasised that they are not predicting the multiple conformations of the functional states of proteins, *i.e.* their structural dynamics.

They were not predicting ligands or bound metals.

They were not predicting complexes of more than one protein subunit.

Progress is being made by different research teams with the fifth and fourth caveats (Perrakis & Sixma, 2021).

Skip forward a year. Accolades are being presented to the DeepMind Team based on their work (Jumper *et al.*, 2021), such as that to the Team Leader, John Jumper, in *Nature*'s Top Ten Researchers of the Past Year 2021 (*Nature*, 2021). A month ago, an article appeared with the startling title "*Structural biology is solved — now what?*" (Ourmazd *et al.*, 2022). This was clearly a stunning commendation of the successes of structure prediction; but this was too much I thought. If not happy with their title, I was quite happy, however, with the reasons for explaining the breakthrough of predicting protein folds from amino acid sequences: "*This delightful success is the culmination of four decades-long efforts: (1) deposition of more than 170,000 experimentally determined protein structures in the openly accessible Protein Databank; (2) deposition of a large number of amino acid sequences of entire families of proteins and their evolutionary relationships in public repositories; (3) elucidation of multiple sequence alignments; and (4) the resurgence of neural-inspired machine-learning algorithms*". This last point illustrates that a new scientific method has indeed reached maturity based on big data; a detailed dissection of the methods used by Jumper *et al.* (2021) is given by Bouatta *et al.* (2021). It is not a hypothesis-driven scientific method. It is also not based on physics and chemistry, which would, as was used for many years, involve setting up force-field equations and arrive at energy-minimised, predicted, protein structures.

Ourmazd *et al.* (2022) went on though to state, abruptly and controversially, I would say: "*Does this success mean that structural biology, as an experimental discipline, is 'solved'? Can we, in good conscience, continue to ask our students and young collaborators to spend months, if not years, determining protein structures? Or is the heyday of protein structure determination finally over? As with any success, it is important to ask what is next. We venture to believe that the full impact of structural biology is yet to come. For, as impressive as these new algorithms are, they cannot predict both protein function and mechanism directly from the amino acid sequence.*" My reaction had been that I could be comfortable with a predicted protein structure, the hypothesis of Tom Terwilliger, if I could refine it against my X-ray diffraction data observations. It would be a game-changer if the model refinement stage were to arrive much more expeditiously than if I had to first solve the protein crystallographic phase problem. Metal content, if any, would be determined by X-ray fluorescence and the identity of the metal atoms and their positions determined by crystallography with tunable X-ray wavelength resonant scattering signals. Ionisable amino acid protonation states would be determined by neutron protein crystallography. Then I would embark on the structure and function stage of research via time-resolved diffraction studies in the crystal or in solution or an ensemble of static crystal structures of a protein, and cryoEM ensembles. The study of the structure and function of 6-phosphogluconate dehydrogenase continues to this day, for example by Hanau & Helliwell (2022).

These observations clearly show that Ourmazd *et al.* (2022) had drawn the boundary between prediction and experiment incorrectly. Terwilliger's recent lecture slide 27 (Fig. 1) shows the likely workflow for protein crystallography in the future, with which I obviously concur (note his starting point at the top left with the *AlphaFold* prediction for each protein chain).

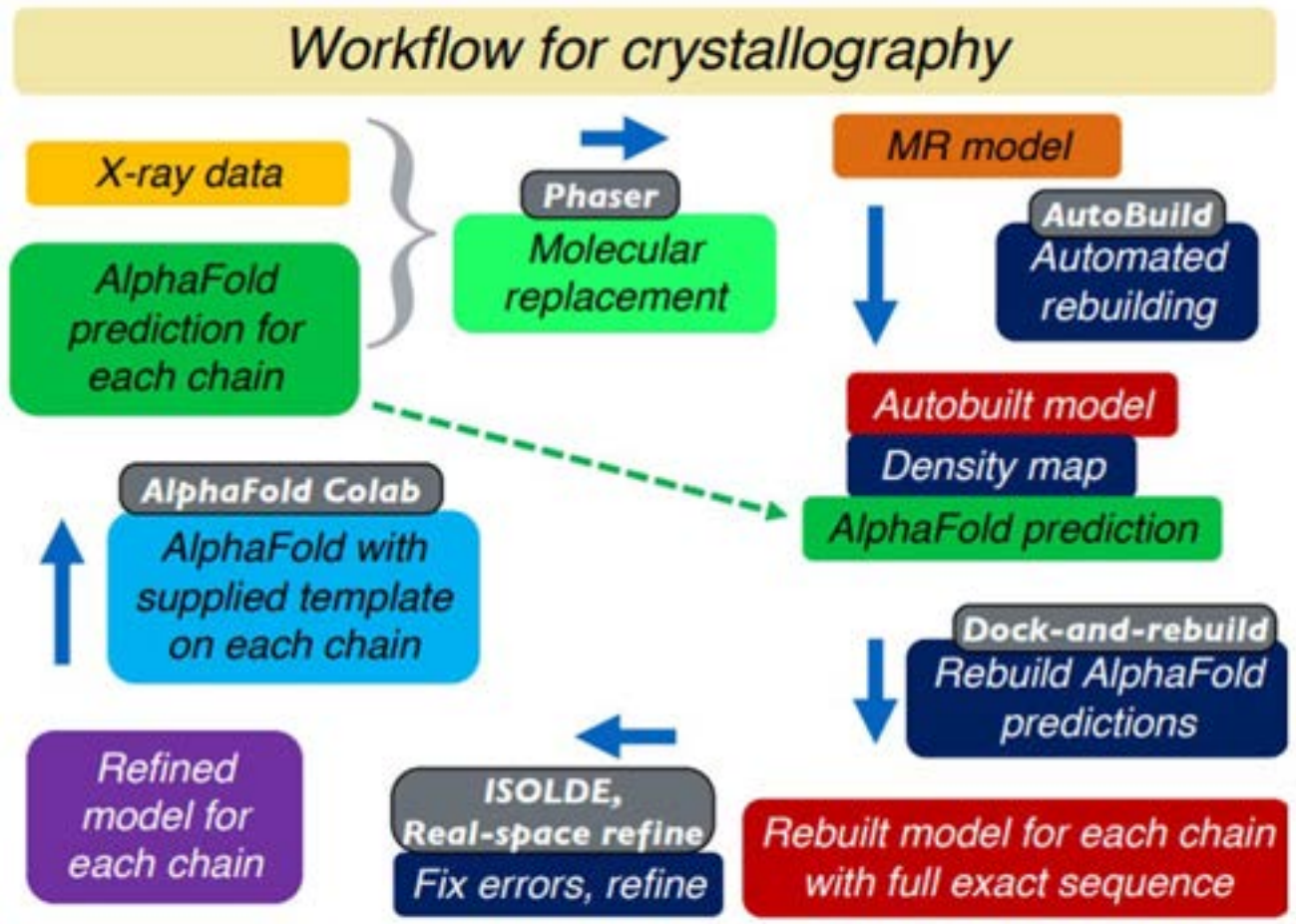


Figure 1. Tom Terwilliger's likely workflow for protein crystallography in the future. The programs listed are within the software package developed by the *Phenix* consortium (Afonine *et al.*, 2012; Liebschner *et al.*, 2019), of which Tom Terwilliger is a member. Reproduced with permission.

A few weeks later, on 4 February 2022, a new Viewpoint appeared in *Science* (Moore *et al.*, 2022), not citing the Ourmazd *et al.* (2022) article but taking the opposite end of the spectrum of views in their article titled "*The protein-folding problem: Not yet solved*". This article includes the assertion that "*At present, for the best cases, the C-alpha coordinate RMSD accuracy of AlphaFold-predicted structures roughly corresponds to the accuracy expected for structures determined at resolutions no better than ~4 Å.*" Furthermore, they state that "*(we) feel that solving the protein-folding problem means making accurate predictions of structures from amino acid sequences starting from first principles based on the underlying physics and chemistry.*"

A point on which we all agree though is well put by Moore *et al.* (2022), who state, "*A further complication for structure prediction is the dynamic structural variation in a given sequence.*"

What about the diffraction resolution for which the *AlphaFold* model can be trusted then? 2 Å or only 4 Å? Taking the sheep liver 2pgd X-ray diffraction data to 2 Å resolution and the human 6-phosphogluconate dehydrogenase predicted structure, the restrained model refinement converges quite normally to an *R* factor of 21%, which is indeed typical. Moreover, the *R* factor versus resolution is shown in Fig. 2 and again is typical.

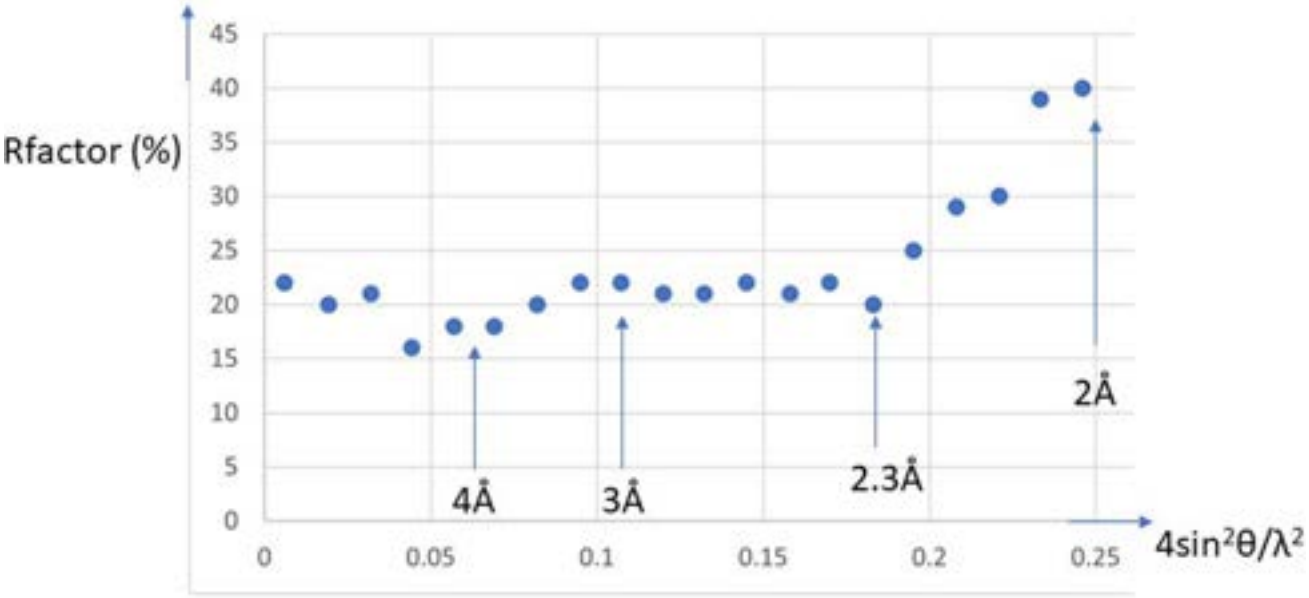


Figure 2. Model refinement in *CCP*4 *REFMAC*5 of the *AlphaFold*-predicted structure hypothesis after placement in the 2pgd unit cell using *Phaser* (McCoy *et al.*, 2007) followed by a rigid body then restrained model refinement in *REFMAC*5 (Murshudov *et al.*, 2011), from which this distribution of *R* factor versus diffraction resolution is given. Specific diffraction resolution points are shown with arrows.

Also note that this set of model refinement statistics is for a model with no bound waters or ions, which *AlphaFold* of course does not predict, and which would improve the *R* factor further. In terms of electron-density maps, Fig. 3 shows the 2pgd model and the *AlphaFold*-predicted structure after model refinement against the 2pgd X-ray diffraction data. The sulfate position and bound waters are placeable without difficulty in this 2 Å high-resolution electron-density map.

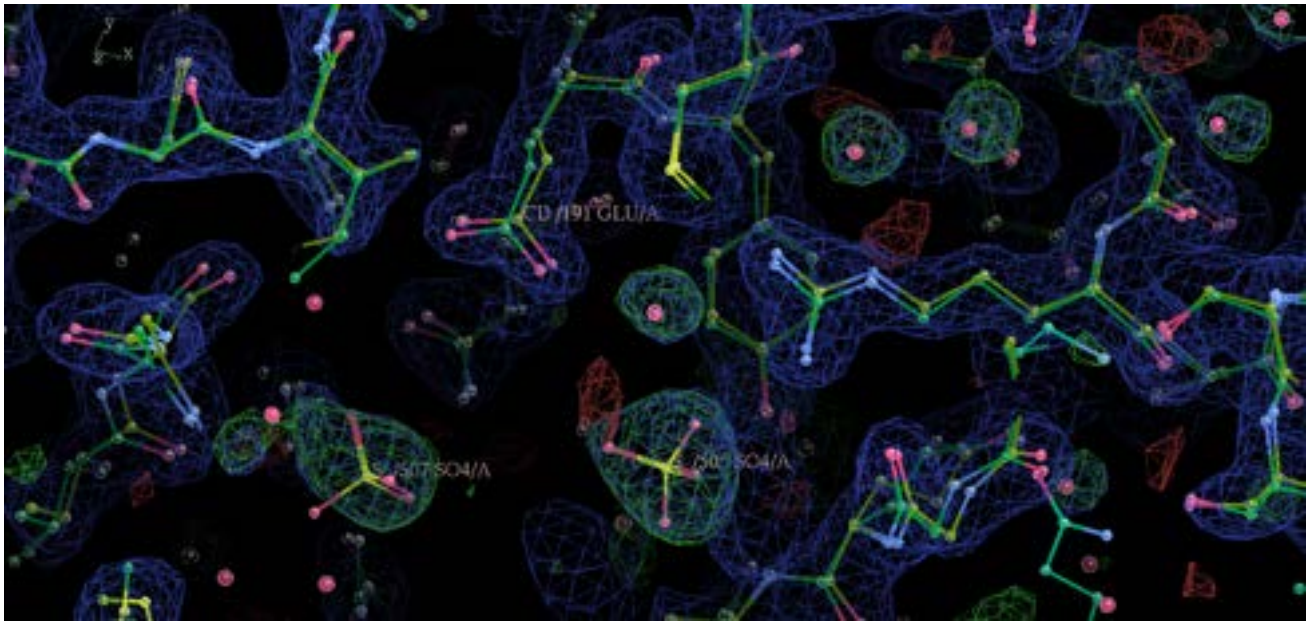


Figure 3. The 2pgd model and the *AlphaFold*-predicted structure after model refinement against the 2pgd X-ray diffraction data. The sulfate position and bound waters are placeable without difficulty. This figure was prepared in the molecular graphics program *Coot* (Emsley *et al.*, 2010).

In summary, *AlphaFold* and *Robetta* will boost the solving of experimental structures by X-rays and electrons. Their models will help those researchers struggling with electron density or electrostatic potential map interpretations of their polypeptides. However, only when we have included in these prediction algorithms the real (or close to real) forcefields, can we really think about having solved the biological structure and dynamics challenges. Nevertheless, the function prediction may well still be left behind unless determined action by funding agencies is taken, which is Ourmazd *et al.*'s main point. Also,

nothing is definitively solved, a lot is still to come and to learn, which is Moore *et al.*'s main point.

Acknowledgement

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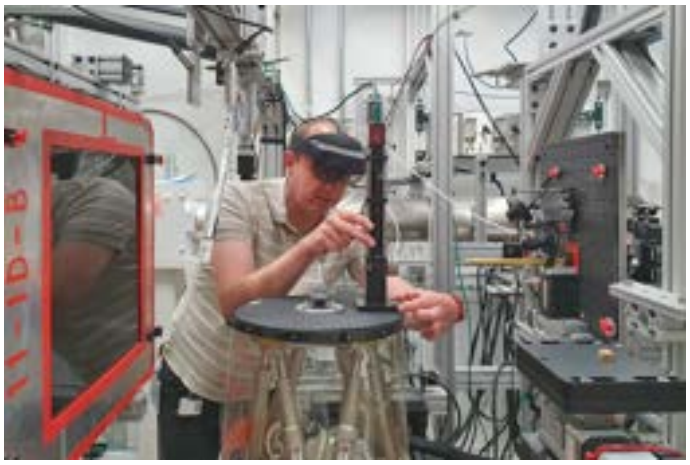
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FEATURE ARTICLE

Enhancing X-ray light source capabilities through augmented and virtual reality: a perspective from the Advanced Photon Source



Olaf Borkiewicz, Mathew Cherukara, Alexis Quental, Brian Rusthoven, Alec Sandy, Kamila Wiaderek, Qingteng Zhang

Driven by dramatic changes in (i) work, home and travel because of SARS-CoV-2; (ii) the increasingly complex nature of experiments that can or will be performed at next-generation X-ray light sources such as X-ray free-electron lasers (XFELs) and multi-bend achromat (MBA) synchrotrons; and (iii) the need to upgrade and operate such facilities with a changed workforce, we see striking opportunities for augmented and virtual reality tools and technologies at X-ray facilities like the Advanced Photon Source (APS) at Argonne National Laboratory. In the remainder of this introduction, we summarize these changes and introduce these technologies at a high level. We also describe emerging and anticipated uses for such technology at the APS and then conclude with our perspective on future opportunities. This contribution focuses on recent work and thoughts at the APS, but we emphasize that many facilities are exploring and implementing similar technologies for the same reasons listed above.

The power and abundance of augmented or mixed (AR or MR) and virtual or meta (VR or MR) devices and capabilities are expected to grow dramatically over the next few years, driven by a confluence of increasingly powerful cloud-computing technologies, advanced wireless capabilities such as 5G and WiFi 6 and increasingly sophisticated edge-computing technologies, especially those powered by artificial intelligence and machine learning (AI and ML). In fact, Statista documents approximately USD 6 billion of consumer spending in the US in 2020 on such technology and an additional USD 2 billion of industrial spending in the same period. By 2024, however, total spending in the US is expected to increase nearly tenfold, with the current consumer–industry split of 75% and 25% expected to flip by that time (*The Economist*).

What are augmented and virtual reality devices?

For those not familiar with the terminology, AR or MR devices project computer-generated imagery or sensory information onto a real-world environment as seen through, for example, a cell-phone screen, automobile windshield or dedicated headset. VR or MR replaces the physical or real world with entirely, or largely, computer-generated sensory information often realized through special-purpose immersive headsets but also found in, for example, flight simulators for commercial or military pilots. Some recent examples of AR and VR are as broad as the Pokémon Go game (AR) that was incredibly popular just a few years ago [Fig. 1(*a*)], the IKEA Place app (AR) that allows consumers to examine virtual furniture in their homes, head-up displays for cars and purpose-built devices such as the Microsoft HoloLens 2 (AR) [Fig. 1(*b*)], Vuzix Blade smart glasses (AR) or Oculus Quest (VR) developed by Facebook [Fig. 1(*c*)]. Facebook has recently rebranded itself as Meta in an indication of its strength and commitment to developing capabilities and tools for virtual reality or the metaverse. Apple and Samsung have also announced plans to release VR devices of their own.

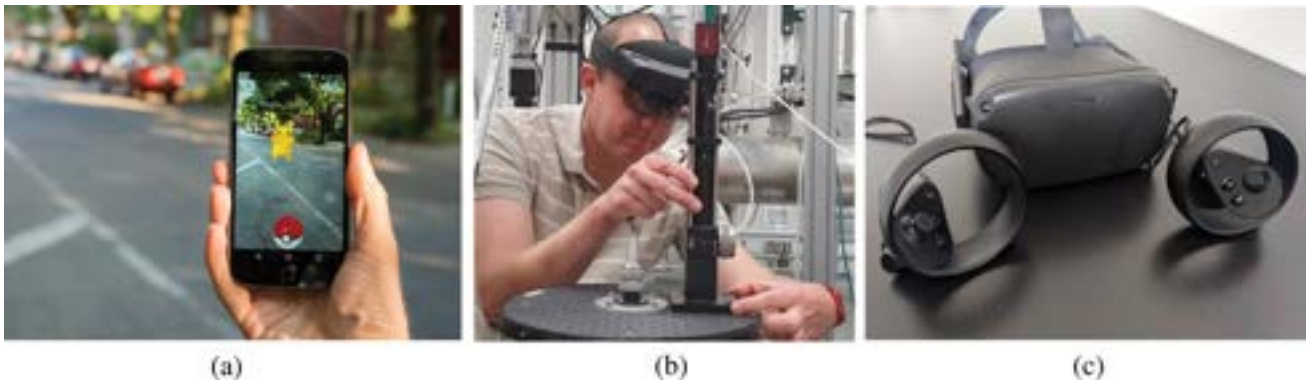


Figure 1. Examples of contemporary AR and VR. (*a*) Pokémon Go (credit: Marc Bruxelle/Shutterstock.com). (*b*) The Microsoft HoloLens 2 being used at 11-ID-B at the APS. (*c*) The Meta Oculus Quest.

Implications for X-ray user facilities

Concurrent with the emergence of AR and VR technology, both the world at large and the world of state-of-the-art X-ray user facilities are undergoing large changes. The past two years or so have brought unprecedented change as the SARS-CoV-2 virus (*IUCr Newsletter*) has continued to play havoc with people's lives and upended our previous notions of work, education and family. The situation with X-ray user facilities like the APS is no different as, traditionally, most work in physical sciences was performed by on-site users who traveled to a user facility for their beam time and worked closely with resident facility staff to perform and interpret their measurements. [This should be compared with macromolecular crystallography (MX), which has been highly automated and with steadily increased remote operations over recent decades (Abola *et al.*, 2000).] Travel and proximity restrictions have made for ongoing challenges, with user facilities switching to a continuum of mail-in and remote access operations augmented by an increasing but still variable and limited on-site presence of users (DOE, 2021). Of course, there are notable exceptions in the physical sciences like mail-in programs for powder diffraction at many synchrotron facilities (Toby *et al.*, 2009), but by and large, prior to SARS-CoV-2, on-site visits by physical science users were the norm. This is not the case now and is not expected to fully revert to the previous arrangement, even after the pandemic (DOE, 2021). Moreover, even as site-access restrictions are eventually eased for staff and users, we believe to expect both greater flexibility and efficiency around work location and site presence. We are confident that AR and VR offer significant opportunities to train and support users and fellow staff members better. Below, we describe in more detail specific directions and opportunities for safety, experimental support and training at the APS.

The science capabilities of X-ray user facilities are also changing markedly, driven by large changes to the facilities themselves. For instance, the APS is undergoing a major upgrade to APS-U that will produce synchrotron X-ray beams hundreds of times brighter than those available today. The APS is currently

scheduled to shut down in April 2023 for a one-year removal, installation and storage ring commissioning project that will result in far brighter beams after completion. The aggressive schedule will require multiple crews working simultaneously in the storage ring tunnel. We see AR with apparatus and location-sensitive training and documentation materials critical to achieving the goals of the APS-U project.

Lastly, significantly brighter X-ray beams are a common deliverable for many X-ray sources throughout the world (Weckert, 2015). We anticipate that lensless imaging techniques (Miao *et al.*, 2015) and lensless imaging combined with other measurements like fluorescence, for example, will produce vast quantities of data and results that will be near impossible to visualize using traditional means. Because of the anticipated scale and complexity, we also think that interpreting data and results from these facilities will require deep collaborations among geographically and scientifically diverse experiment teams. We are confident that AR and especially VR will prove invaluable in this regard.

The remainder of this overview is organized as follows. First, we discuss recent work with AR to support user experiments. Second, we discuss opportunities and work in progress for AR to support installation, maintenance and design activities. Third, we discuss some of the possibilities for AR and VR to enhance data visualization, help with outreach and education, and further collaboration among geographically diverse experiment teams. Finally, we offer our perspective for longer-term opportunities. We note that, as all the authors of this article are from the APS, our examples and perspective are largely confined to one AR device on which we have focused our investments and effort to date, namely the Microsoft HoloLens 2. We have been impressed with this device's capabilities to meet our needs but this article should not serve as an endorsement, and we encourage anyone interested in the technology to conduct their own evaluation of which AR and VR platforms best meet their requirements: there are many on the market and more forthcoming.

Using AR to support user experiments

The onset of the SARS-CoV-2 pandemic and the subsequent switch to an increasingly remote work paradigm have upended the classical workflow at synchrotron beamlines worldwide. This is especially true for *in situ* and *operando* measurements that involve complex setups and integration of various experiment-specific equipment with beamline infrastructure. The successful execution of such experiments relies heavily on multiple aspects of preparation and operation but arguably the most important is clear communication and close interaction between the beamline personnel and users. Lockdowns and site access restrictions in response to the pandemic initially made carrying out these types of experiments impossible. As a first step, the APS, like many user facilities, developed and provided capabilities so that remote operation of a beamline and experiment was possible by both users and staff. This included the addition of equipment used to support remote operations, such as internet-enabled cameras inside and outside hutches, remote access to beamline computers, robotics and automation, and the use of various communications platforms. Nevertheless, the quality of support that beamline personnel could offer to remote users was far from ideal.

The emergence and popularization of AR devices has significantly changed the way experiments – both remote and on-site – are being set up and carried out at beamlines. Implementation of the Microsoft HoloLens 2 at APS beamlines, for example, has opened new ways for beamline scientists to communicate and interact with their remote users. The first-person perspective offered by the device is valuable for both the device user and the remote user. The head-mounted camera offers outstanding image quality and allows for unrestricted hand movement. This makes regular beamline tasks, such as building experimental setups, sample manipulation, assembly and disassembly of sample environments *etc.* not only easier but also much safer. In addition to allowing for a seamless, constant two-way communication between the device operator and the user, the device offers the capability of real-time annotating of the so-called holo-field (field-of-view for both parties). This provides additional, non-verbal levels of communication that are extremely helpful in troubleshooting a problem or in simply explaining the details of tasks being undertaken. As examples, Fig. 2 shows screenshots that capture the first-person

view seen by the on-site staff person providing electrochemical-cell assembly support for a remote participant.



Figure 2. Screenshots of the first-person perspectives provided by the HoloLens 2 during the preparation of AMPIX (electrochemical) cells within a glovebox at the APS Electrochemical Facility. In (*a*), a holographic sample list is visible in the holo-field.

The device has also changed the way resident staff provide out-of-hours user support. Until recently, in the event of a failure or experiment problem at the beamline, users were limited to telephone or videoconference communication with the support staff, and the beamline scientists were forced to troubleshoot the problem via the same channels. In the case of a more complex issue, a resident staff member would have to return to the facility to resolve the issue. With an AR device, the complexity of issues that can be solved remotely has increased significantly. In addition to being deployed at the beamlines, the HoloLens 2 device has been used by the APS Electrochemical Facility for the preparation of the samples for electrochemical experiments at various beamlines of the APS. The first step of these experiments requires the assembly of a custom-made testing cell consisting of several small pieces: this work needs to be performed within an inert atmosphere of a glovebox. Because of the complex nature of the assembly process and restrictions related to working within the glovebox, it was virtually impossible for users to follow the preparation using traditional videoconferencing tools. The AR device provides a first-person view of the work in the glovebox [Fig. 2(*a*)] and the remote users greatly appreciate the ability to follow every stage of the preparation while remaining in real-time communication with the scientist assembling the cell. Another feature of AR devices like the HoloLens 2 is in mixed-reality applications that assist device operators with work by providing holographic instruction in the form of documents, images, videos or 3D holographic models. Instructions are triggered by 'anchors' strategically placed in relevant beamline areas or near equipment. In this example, the scientist preparing the samples can project holographic sample preparation instructions and display them on the virtual screen in their field of vision [Fig. 2(*a*), white 'paper' in the top right of the image]. This feature greatly reduces errors and simplifies the execution of the work.

Using AR to support installation, maintenance and design activities

The APS is undergoing a USD 815 million project funded by the DOE to replace its third-generation synchrotron storage ring with an MBA storage ring (many beamline replacements and upgrades are also included in the scope of the project). The project will remove all the existing equipment from the 1.1 km storage ring tunnel and environs and replace it with the latest technology. The APS storage ring, both before and after the upgrade, has 40 sectors. Many sub-assemblies comprise the infrastructure in each sector. Each sub-assembly consists of about a dozen magnets, a dozen vacuum chambers with support systems, absorbers, valves and crosses, each with their own support systems, water supply and return headers with branch tubing for each magnet and vacuum chamber, along with required grounding bus bars and grounding straps.

To accomplish the storage ring rebuild in the short one-year timeline for removal, installation and initial commissioning, the APS is increasing its (temporary) technical staff. This has led to training and support needs that conventional media cannot meet. Instead of requiring Subject Matter Experts (SMEs) to walk

each new hiree through the technical requirements of an assembly, we have developed instruction sets that will train these new employees using a workflow application that is available through the HoloLens 2 interface. This approach will allow the SMEs to advance the training and support of new hirees without overburdening the SMEs. Fig. 3 displays screenshots from a HoloLens 2 showing some of the real-life sub-assemblies being assembled with instructions and visual cues provided by AR overlays.

After the APS upgrade is complete and we move into operation of the new facility, we anticipate AR assisting in the future maintenance of the storage ring. For example, maintenance procedures for each magnet type require many complex tasks that can be documented and then referred to by technicians while they perform the work. This opportunity will ease the transfer of information from senior staff to the next generation of SMEs, keeping the APS a world-class machine far into the future.



Figure 3. APS upgrade storage ring sub-assemblies viewed through a VR headset. The view is augmented with virtual models and instruction sets to aid in the assembly and maintenance of the components shown that will soon be part of the APS storage ring.

In addition to the storage ring installation and maintenance activities described above, we see additional opportunities for AR and VR to assist in X-ray beamline debugging and maintenance. The Component Database (CDB) is an application that has been designed and built to aid in documenting and managing all the parts that will go into APS-U. The CDB allows design information about the storage ring and beamline components to be retrieved quickly using a QR code that can be scanned using an AR device. With this information, beamline staff and engineers will visualize the digital twin alongside its physical counterpart while simultaneously displaying a dedicated hologram panel of records including technical parameters, procurement status and use history.

Fig. 4 shows an example of enhanced perception and record-keeping using a digital twin enabled by an AR headset. The metallic piece in the transparent plastic box in Fig. 4(*a*) contains a cylindrical channel that leads to an inner circular chamber. This structure resembles enclosed beamline components (*e.g.* monochromator, vacuum sample chamber *etc.*) whose inner structures cannot be easily examined, and where CAD drawings of one or all components may not be readily available or accessible. By scanning the QR code printed on the box, the HoloLens user can open the digital twin of the physical component from within the CDB, where a full record of the component is available [Fig. 4(*b*)]. In addition, the digital twin contains a hologram of the physical object that can be visualized next to the physical object for enhanced perception [Fig. 4(*c*)]. It is worth noting that with the HoloLens, the contour of all inner structures becomes visible when the hologram is viewed from inside [Fig. 4(*d*)].

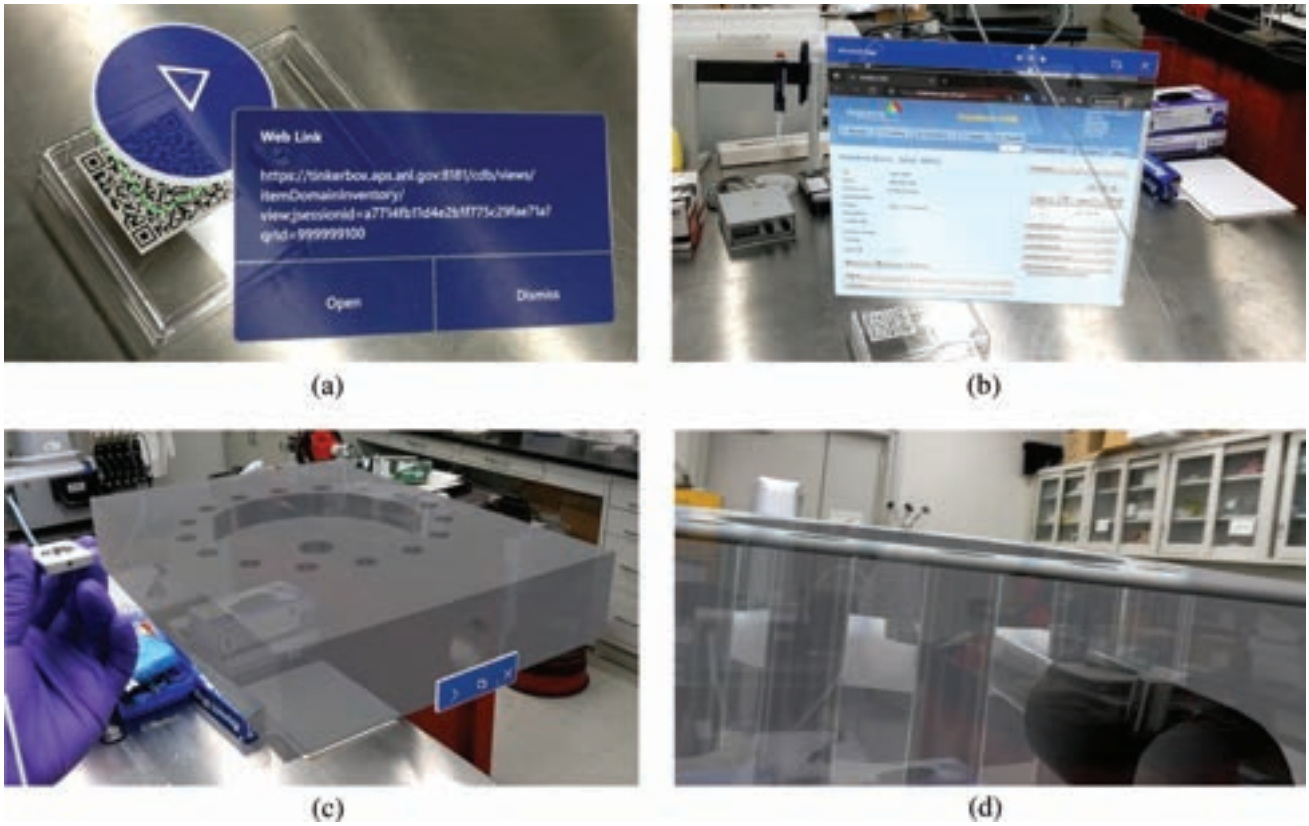


Figure 4. (*a*) and (*b*) Pairing a beamline component with its digital twin via QR-code scanning. (*c*) and (*d*) Visualization of the digital twin in holograms on an AR headset.

Using AR and VR to enhance data visualization

An increasing number of instruments at light sources generate high-dimensional data. These include instruments performing various 3D imaging techniques or from the simultaneous acquisition of multimodal data. Interacting with and extracting meaningful scientific insight from such datasets is challenging when data can only be visualized in 2D. AR devices provide a natural way of interacting with 3D data and provide more flexibility with even higher-dimensional data. In addition, to enhance human–data interaction, AR devices can increase the efficiency of scientific collaboration. Two people each wearing an AR device can simultaneously view a hologram along with remote collaborators. Fig. 5 shows an example of visualizing 3D imaging data with the Microsoft HoloLens 2.

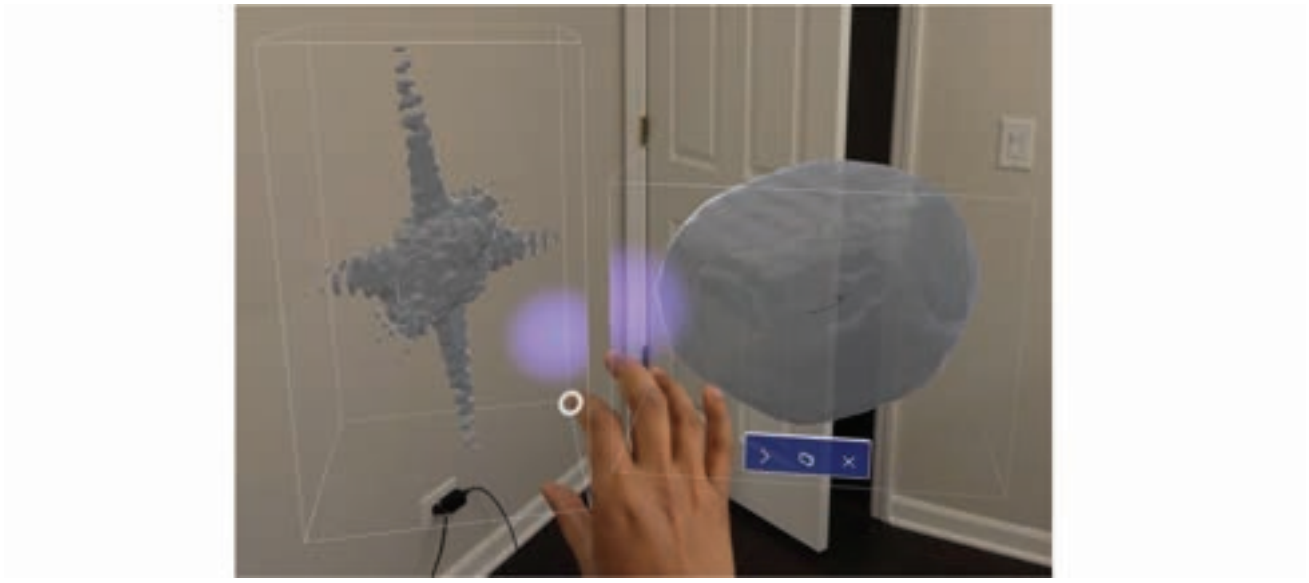


Figure 5. Example of visualizing 3D imaging data with the Microsoft HoloLens 2. The left hologram is the 3D Bragg Coherent Diffraction Imaging (BCDI) pattern assembled from many X-ray measurements in reciprocal space, and the right hologram is the reconstructed (real space) faceted nanoparticle determined via phase inversion techniques applied

to the measurements.

Using AR and VR to help with outreach and education

The HoloLens 2 device has also proven to be an essential tool for continuing educational programs at the APS during the pandemic. We have used it as a telepresence tool for remote students, with the most notable examples being the National X-ray School on Neutron and X-ray Scattering (NX School) and Argonne's Exemplary Student Research Program (ESRP). In the pre-pandemic era, these programs offered graduate-, college- and high-school-level students unique opportunities to design, work on and conduct hands-on experiments with scientists at the APS. The pandemic has interrupted the decades-long tradition of hosting users on-site, but the HoloLens 2 provided an immersive experience for the participants. In both cases, scientists from Argonne hosted virtual experiments and used the AR devices during the experiments to augment online lectures.

Longer term opportunities

While still a nascent technology, AR devices have already made a mark on the user experience at the APS, and with significant investment from industry, we can expect to see the capability and versatility of these devices grow. Future AR devices that provide more processing power and an expanded software ecosystem can further enhance beamline experience and productivity. One such avenue could be through making human–machine interaction more efficient. Modern beamlines at synchrotron and XFEL sources are extremely complex instruments with a great many control variables. A typical beamline desktop has windows for dozens of motor controls required for standard operation that can be overwhelming to new users. Rather than a non-intuitive 2D array of motor controls, users could use 3D holograms of the beamline to target and move motors, run scans *etc*. through hand gestures. We are working on an in-house application that can be used to view the current state of the equipment inside the experimental hutch. This is the first step in further facilitating setup changes by both staff and users. The goal is to keep the framework flexible and low maintenance. In this way, the staff can accommodate modifications to the existing equipment without having to completely re-do the programming. An early mock-up of such a display is shown in Fig. 6. Positioner names are listed on the left of the display and their current values to the right.

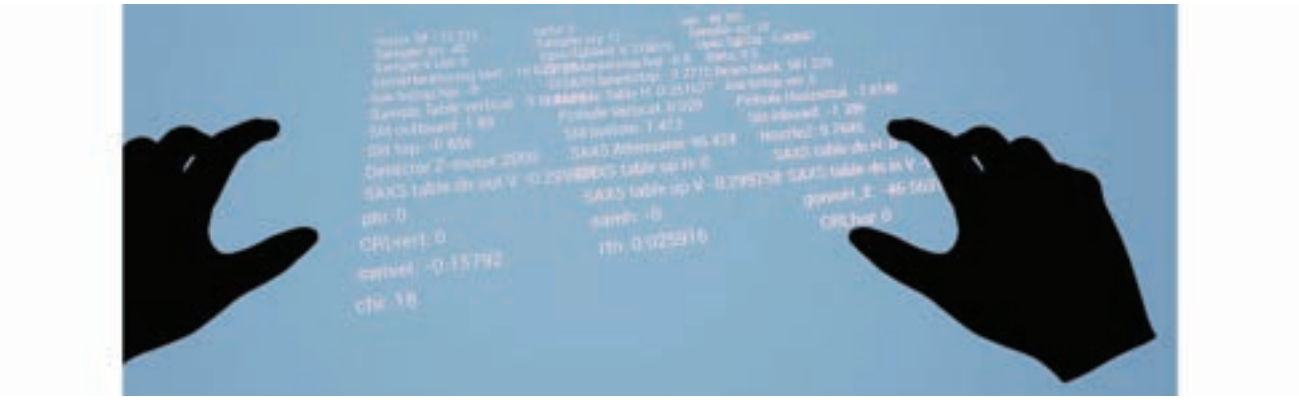


Figure 6. An experiment station's motion device names and values displayed as a table. The left side of the image shows a station's positioner names while their positions are displayed on the right.

Looking further ahead, by combining visualization with gesture-controlled scanning, users could simply identify regions of interest in high-dimensional data, select the appropriate scan ranges from the display and launch follow-on scans much faster and more intuitively than possible with a conventional computer.

Acknowledgements

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FEATURE ARTICLE

Machine learning in crystallography

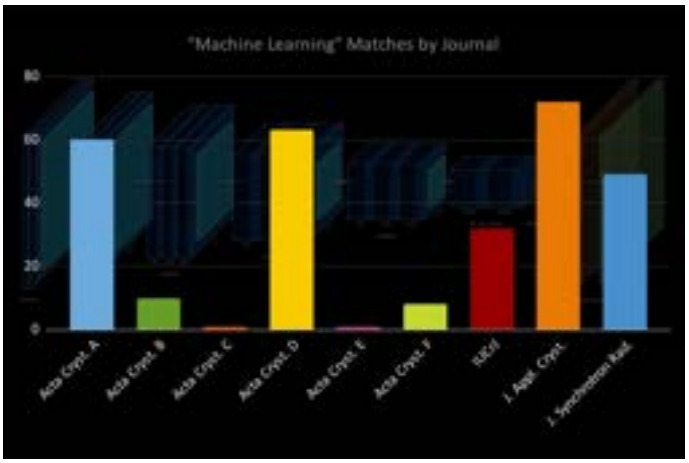


Figure 1. Number of articles matching the search term 'machine learning' by IUCr journal. Data compiled from site https://journals.iucr.org/ (15 February 2022).

Thomas Proffen

By now, everyone has probably heard the term 'machine learning'. We have all used tools in our everyday lives that use machine learning, from voice assistance to image recognition to ever more sophisticated algorithms showing us online ads of what we supposedly want to buy. There have been great advances in medicine based on machine learning, especially in the area of radiology. As more of our lives are affected by this new technology, the question of bias and the limits of its application have come up – an interesting read on these aspects is Cathy O'Neil's book *Weapons of Math Destruction* [1].

The question in many researchers' minds is what is the impact of machine learning and artificial intelligence (AI) going to be on their field of science – in this case, crystallography. A quick full-text search of the term 'machine learning' in all IUCr journals reveals 296 articles (as of February 2022). While this is clearly a non-comprehensive survey of crystallographic papers using machine learning in some form, and this simple full-text search has its limitations, a quick breakdown by journal (Fig. 1) gives an interesting perspective on the breadth of areas within crystallography on which machine learning has an impact.

Interestingly, the first paper in this search was published in 1993 in *Acta Cryst. D* on integrating direct methods and AI strategies for solving protein structures [2]. Structural biology has remained a field active in exploring machine learning and AI in order to move forward. However, across nearly all the journals and the related fields, articles exploring how machine learning and related techniques can be applied go back 20+ years. Browsing through the list of papers was an excellent way to bring up ideas from work in a completely different context and made me appreciate the breadth of crystallography as a discipline.

One approach is so-called supervised machine learning, where labelled data are used to train a neural network that is then used for classification or regression of other data. Image recognition is a prime example. Once you train a neural network with enough labelled images (*i.e.* the computer gets told what is on the image) of cats and dogs, the network will be able to identify cats and dogs on other images. Because we can often calculate the signal we expect from an experiment based on a model, we can create training data through simulation and use the model parameters as labels. This allows a speed up of expensive calculations by having the neural network 'interpolate', but it can also be used to, for example, determine the space group from diffraction and pair distribution function data [3, 4]. This method of classification also opens the door to machine-learning-generated metadata, for example to

label experimental data as 'good', 'bad', 'misaligned' and so on. While these labels might need verification by a scientist, this approach offers an easy way of flagging problems, especially on high-throughput instruments.

One of the recent exciting developments is that machine learning has become much more accessible, thanks to advanced and easy-to-use tools developed by the major tech companies. This gives us access to machine-learning libraries such as TensorFlow [5] or PyTorch [6] and easy-to-use tools requiring virtually no coding. I am involved in Oak Ridge Computer Science Girls [7], and we teach middle-school girls about machine learning using Teachable Machine [8]. Here, they can train a neutron network to classify images. The impact of these tools became evident in a session on AI at the 2021 ACA meeting, when Scott Classen from the Advanced Light Source Berkeley presented machine learning aiding sample centering and included a 'behind the scenes' look of how the tool was built using essentially a Google Cloud Drag and Drop tool [for details see 9]. In the same session, Dan Olds, from Brookhaven National Laboratory, presented an in-depth overview of the many areas in which machine learning and AI will have an impact on user facilities in accelerating scientific discovery and making using a beamline more streamlined. He introduced a novel approach to use so-called reinforcement learning to have the system 'learn' what a good experiment looks like [10].

In summary, there is a lot of exciting work involving machine learning in the crystallographic community from structure solution to data analysis and running smarter experiments. In many ways, this is just the start. So will machine learning replace crystallographers? No, but crystallographers using machine-learning techniques will likely surpass those who do not.

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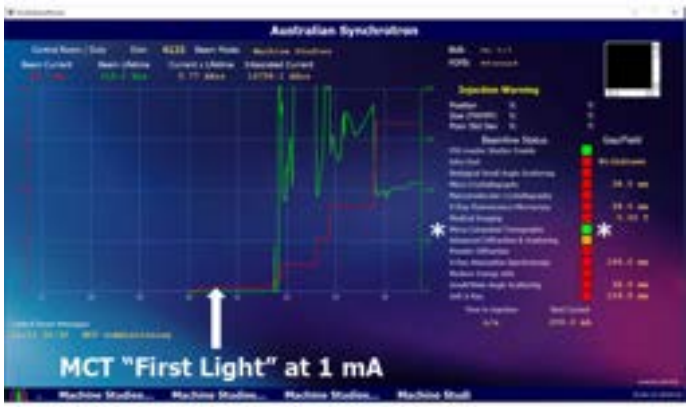
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FEATURE ARTICLE

A bright future at the Australian Synchrotron



Facility Status Monitor showing “First Light” on the Micro-Computed Tomography Beamline (image courtesy of the Australian Synchrotron).

Christopher Sumby, with contributions from Michael James

The Australian Synchrotron is classified as one of Australia’s “landmark pieces of scientific infrastructure,” serving over 7000 researchers from around the world but predominantly from Australia, New Zealand and the Asia-Pacific region. The users come from a mix of universities, medical research institutes, government and other research organisations, and industry. This year notably marks 15 years of operation and user access.

The facility has 10 original beamlines in operation, but the current BRIGHT Program[1] is investing nearly $AUD 100 million in a near doubling of the number of the beamlines. This includes the construction of eight additional beamlines covering imaging, spectroscopy, microscopy, diffraction and scattering experiments. The aim of this expansion is to meet the needs of Australasian researchers and industry well into the future and to usher in new capabilities and provide access to new experiments needed for breakthrough research.

The BRIGHT Program was initiated in 2016 following on from the commitment of the Australian Commonwealth Government to provide operational funding of the facility for 10 years through to mid-2026. Over the next couple of years, around $AUD 100 million investment was secured under the BRIGHT Program from more than 30 Australian and New Zealand research organisations to cover the construction costs of the eight new beamlines.

Over the last few years, significant planning, extensive consultation with user groups, design, procurement and construction components of the BRIGHT beamlines have been taking place, much of it against a background of the COVID-19 pandemic. Despite these challenges, a significant milestone was achieved in late 2021 with “first light” achieved on the Micro-Computed Tomography (MCT) beamline, as shown in the headline image.



Views of the Advanced Diffraction and Micro-Computed Tomography beamline hutches under construction (photo courtesy of Michael James, Australian Synchrotron).

As each beamline has been given approval, the beamline has gained a team responsible for the beamline design, build and future operations, under the stewardship of an experienced Lead Scientist, working in partnership with a Project Manager and a Lead Engineer as part of an integrated project team. These teams are responsible for the individual beamlines and work with the overall Australian Synchrotron BRIGHT Program team.

The planning, design and construction of the new beamlines does not take place in isolation. Existing and potential new users are consulted through fora such as conferences, workshops and user panels. The design of the beamlines is reviewed by beamline scientists at other synchrotron facilities, by the broader in-house team and by the facility's Scientific Advisory Committees. Moreover, a Beamline Advisory Panel (BAP) is in place for all beamlines, headed by a local scientist of significant international repute who is an expert in the area and involves expert users and beamline scientists from other facilities. Extensive expert review and detailed modelling of the beamline design is conducted well before any construction work and equipment procurements go out to tender.

The final eight new beamlines include Micro-Computed Tomography (MCT), High Performance Macromolecular Crystallography (MX3), Advanced Diffraction and Scattering (ADS-1 and ADS-2), Biological Small Angle X-ray Scattering (BioSAXS), X-ray Fluorescence Nanoprobe (NANO) and Medium Energy X-ray Absorption Spectroscopy (MEX-1 and MEX-2) beamlines, offering a mix of new techniques and capabilities, different accessible energies, and studies of new types of samples in different environments. While the *IUCr Newsletter* readers will have diverse interests, the rest of this article focuses predominantly on the beamlines in the areas of X-ray absorption, scattering and diffraction.

X-ray absorption spectroscopy

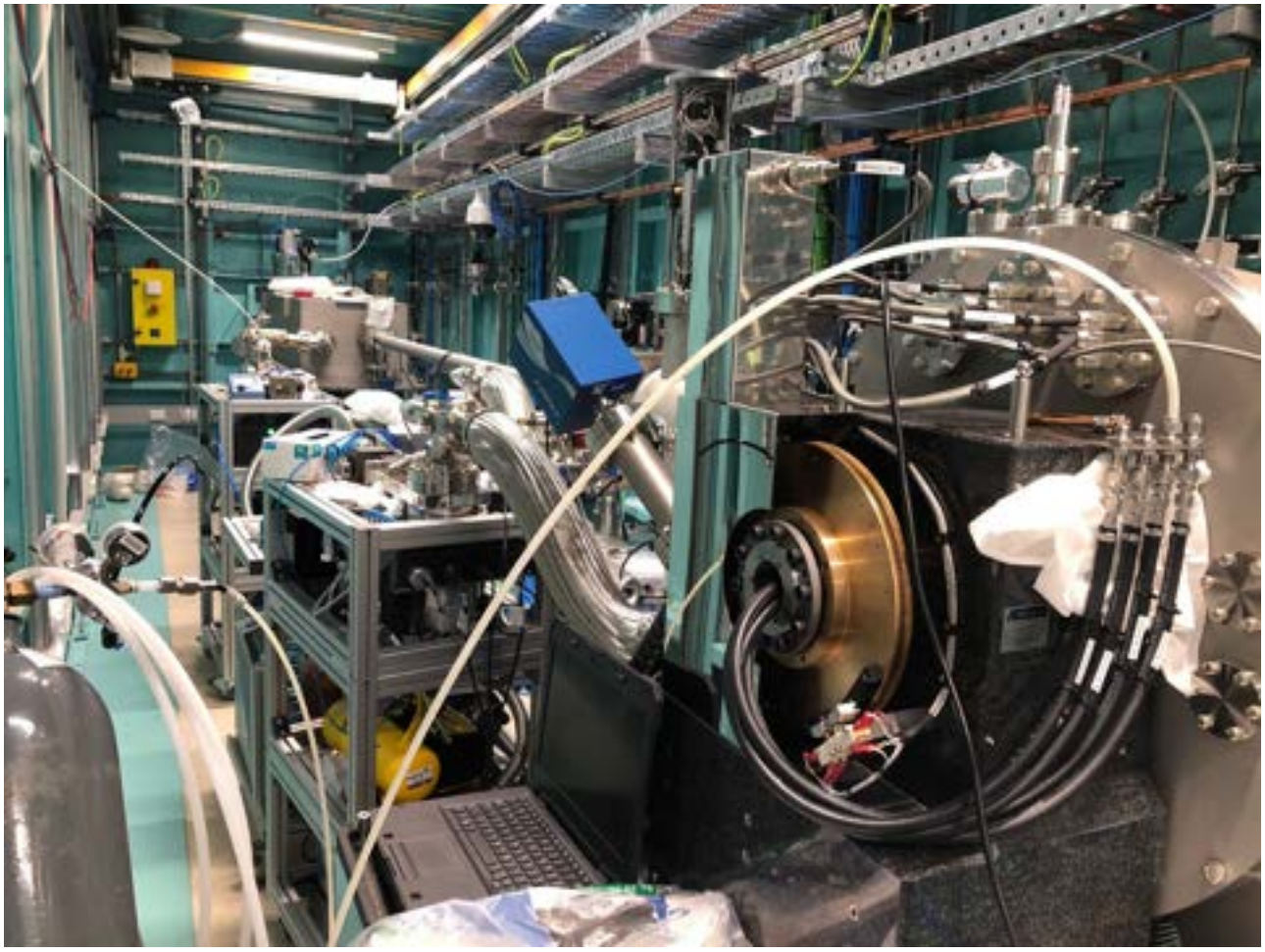
Two of the new suites of beamlines are the Medium Energy X-ray Absorption Spectroscopy (MEX) beamlines (MEX-1 and MEX-2). These will provide X-ray absorption spectroscopy (XAS) capabilities to map the chemistry and element distribution within materials. While the existing wiggler-based XAS beamline at the Australian Synchrotron is designed for heavier metallic elements, the MEX beamlines can target the lighter biologically relevant elements (Si, P, S, Cl, K and Ca) and transition metals with less impact from radiation damage to the samples. MEX-1 and MEX-2 are well-suited to applications in health, agriculture, environmental monitoring, energy and geosciences as well as museum and cultural heritage studies. The MEX beamlines use a bending magnet source, with an energy range intermediate to the wiggler and undulator sources in the current XAS and Soft X-ray Spectroscopy beamlines. Whereas the bending magnet source is common to both MEX-1 and MEX-2, these beamlines will be able to operate independently and simultaneously. MEX-2 will operate with a lower-energy X-ray window (1.7–3.5 keV) with samples housed under vacuum or a helium atmosphere, and the ability to vary sample temperature from 10 to 500 K. MEX-1 will operate in the “tender” X-ray window (3.5–13.6 keV), where samples can be measured under ambient conditions or under cryostat conditions down to 10 K. MEX-1 will operate under a range of different modalities: transmission or fluorescence XAS, high-energy resolution mode using a crystal analyser spectrometer, and in a scanning microprobe mode (with 2–8 micron resolution). The vast majority of equipment for the MEX beamlines is onsite and undergoing testing. Installation of beamline systems will continue throughout 2022, with “first light” expected mid-year and the commencement of the user program in early 2023.

Macromolecular crystallography

The High Performance Macromolecular Crystallography (MX3) beamline is an in-vacuum undulator beamline being built to complement the current MX1 (bending magnet) and MX2 (in-vacuum undulator) beamlines. Somewhat differently to other international synchrotron facilities, the crystallography beamlines at the Australian Synchrotron handle both chemical and biological (protein) macromolecular samples, with access granted based on the sample requirements and beamline capabilities rather than sample type (biological versus chemical). The plan for the MX3 beamline is to provide a very high-flux, micro-focused X-ray beam (down to 2 x 2 microns) for very small and weakly diffracting protein crystals (mainly), along with an emphasis on high-throughput protein crystallography with significant automation. There are also planned capabilities for in-tray screening and serial crystallography. Orders for all the major systems for MX3 have been placed, with the commencement of onsite installations to take place in 2022.

Scattering and diffraction

The Australian Synchrotron currently has a Small and Wide Angle X-ray Scattering (SAXS/WAXS) beamline, but will add a new solution scattering-focused Biological Small Angle X-ray Scattering (BioSAXS) beamline. This will combine a superconducting undulator with a double multilayer monochromator to produce a very high brilliance X-ray beam over a narrower energy range that will be suitable to enable millisecond dynamics studies to take place. The BioSAXS beamline will support high-throughput and highly automated solution scattering experiments from chemical and biological systems and allow complex sample environments (rheometry, stopped-flow, micro-fluidics, applied magnetic fields *etc*.) for studies of liquid phase systems, increasing the capacity of the existing highly used SAXS/WAXS beamline. A protein autoloader based on the CoFlow method[2] will be implemented in concert with size exclusion chromatography to enable high-throughput protein solution scattering while minimising radiation damage. The installation of BioSAXS systems is well advanced, with “first light” expected mid-2022 and first users in early 2023.



The BioSAXS hutch showing the optics installation taking place (photo courtesy of Michael James, Australian Synchrotron).

Finally, the Advanced Diffraction and Scattering beamlines (ADS-1 and ADS-2) will allow high-energy diffraction (up to 150 keV), pair distribution function (PDF) analysis, Laue diffraction, energy-dispersive diffraction imaging and time-resolved and extreme environment powder diffraction. These two beamlines will complement several other beamlines in the facility, being capable of undertaking single-crystal diffraction studies, especially in non-standard sample environments (supporting MX1, MX2 and MX3), and strain scanning and probing of bulk materials (complementing the Powder Diffraction and Imaging and Medical beamlines). The ADS-1 endstation will be housed in a purpose-built satellite building to support industry-relevant studies and the measurement of large, engineered components (up to 300 kg), while the ADS-2 endstation will operate within the main Synchrotron Building. The two beamlines will be able to operate concurrently with multiple sample configurations and environments using high-energy mono- and polychromatic X-rays. This will allow industrial and academic studies of mineral formation under extreme conditions; analysis of large, manufactured objects; failure studies of engineering infrastructure; and characterisation of complex multi-component systems, such as battery and fuel cell devices. Construction of the ADS-1 Building has commenced, along with the installation of the ADS radiation enclosures. Installation of the remaining insertion device and beamline systems will take place over the next 12–18 months, with “first light” expected in mid-2023 and first users in early 2024.

The BRIGHT Program has operated throughout the COVID pandemic, requiring new ways of working for the new beamline teams and interactions with external vendors, especially those located overseas (remote and virtual component testing *etc*.). “The COVID experience in Victoria made the challenges even more difficult than they would be in a normal beamline construction project,” commented Senior Principal Scientist Dr Michael James. “Because the numbers of support people who could come from overseas vendors were limited, our staff have had to take on extra responsibilities,” he added.

All the proposed beamlines are in various stages of planning, procurement, construction or commissioning for user operations. The progress of this exciting new phase of the Australian Synchrotron can be followed via the near-monthly updates published on the Australian Synchrotron webpage (https://www.ansto.gov.au/our-facilities/australian-synchrotron/project-bright).

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STATEMENT

ECA condemns the invasion of Ukraine



The European Crystallographic Association (ECA) is joining a large and growing number of European and international research organizations in condemning the acts of aggression by the armed forces of the Russian Federation and the military invasion of its independent neighbor, Ukraine, a national member of the ECA.

Our thoughts are with our fellow crystallographers, their families, friends, and the people of Ukraine, whose daily suffering is beyond imagination. At the same time, we applaud and stand in solidarity with those Russian scientists who courageously speak out against this unlawful military aggression in their home country and elsewhere, and trust they will not be impacted by expressing their human right to do so. Moreover, we appeal to local, national, European, and world authorities to make every effort to restore peace immediately, before more innocent civilians are severely impacted or lose their lives. Diplomacy, dialogue, and goodwill are the means for resolving serious conflicts such as the present one, and we call for these.

During 50 years of concerted scientific activities, the European crystallographic community has provided a platform for the exchange of scientific knowledge across national borders and cultures. The ECA is committed to continue its efforts to remain a resource to foster future collaborations and peaceful and sustainable coexistence.

In 2021, the Council of the ECA invited the city of Lviv to host the European Crystallographic Meeting (ECM35). Along with all crystallographers in Europe, we are looking forward to celebrating this event in 2025 in a peaceful, free, and flourishing Ukraine.

SPECIAL REPORT

Crystallography in France, a selective instant view



Pierre Bordet, Arie van der Lee, Joanna Timmins, Etienne Balan, Delphine Cabaret, René Guinebretière, Matias Velázquez, Philippe Guionneau

Foreword

Academic scientific research in France is based around 70 universities, each with numerous departments, sites and branches, as well as around major research organisations. These include not only the Centre National de la Recherche Scientifique (CNRS), recognised as the second most important research institution in the world (Scimago Institutions Rankings), but also the Commissariat à l'Énergie Atomique et aux Énergies Alternatives (CEA) and the Institut National de la Santé et de la Recherche Médicale (INSERM). In this context, and given the multidisciplinary nature of crystallography, it would appear complex, if not impossible, to draw up a complete and exhaustive overview of the activity of this science in France. This is not the purpose of this article. Nor is it the purpose of this article to relate the importance of the past activities of French researchers in crystallography. We will therefore not talk about Auguste Bravais, Georges Friedel, René Just Haüy or Charles Mauguin, nor about Louis Pasteur or the French crystallographers who were President of the IUCr such as André Guinier, Jean Wyart and André Authier. That is another story. The purpose of this opus is to illustrate the current status and dynamics of crystallography in France by focusing on a few major facts. By definition, the result is partial and probably also unintentionally biased. May the reader excuse us for this. However, despite the inherent flaws of the exercise, an initial mapping of our science in France is sketched out and testifies to a certain dynamism and a swarming of ideas and results, fruits of the hard work of so many colleagues. The purpose of this article is to pay tribute to them as France is preparing to host major crystallographic events in 2022: the 3rd European School and the 7th European Conference on Crystal Growth, July 2022, Paris; the 23rd General Meeting of the International Mineralogical Association, July 2022, Lyon; and the 33rd European Crystallography Meeting, August 2022, Versailles.

1. The French constellation at a glance

On the French territory (Fig. 1), the crystallography community is, in the first place, represented and animated by the French Crystallographic Association (AFC for Association Française de Cristallographie). This is organised around thematic groups of activities (Biology, Chemistry, Physics) and related subjects (crystal growth, large-scale instruments, teaching and scientific mediation). Crystallographers close to mineralogists are also grouped together in the Société Française de Minéralogie et de Cristallographie (SFMC) and in the Groupe Français des Argiles (GFA). More broadly, a constellation of associations and networks covers the numerous interfaces between crystallography and other disciplines, techniques and know-how. For example, the Société Française de Neutronique (SFN) and the Société Française de Biophysique (SFB) promote the interfaces between crystallography and the corresponding disciplines, respectively neutronics and biophysics. We should also point out that there are very active research networks in various crystallography fields, such as structural crystallography, crystal growth and structural biology, with the ЯECIPROCS, CRISTECH and ReNaFoBiS networks, respectively. In some cities, local associations of researchers further promote crystallography on university campuses and towards a wider public. This is the case in Marseille, Bordeaux, Grenoble and Strasbourg, for instance.

French crystallographers meet regularly at national congresses and at more specific colloquia. As everyone knows, crystallographers like to take the parity of a number into account! In even-numbered years (except for pandemic disruptions), the AFC general congress always gathers more than 200 participants [last editions: Marseille (2016), Lyon (2018), Grenoble (2021)]. This figure is also attained in odd years with the Integrative Structural Biology Meeting [Toulouse (2019), Paris-Saclay (2021)], which attracts numerous biologists from the community, and the X-Rays and Matter Conference [Nancy (2019), Aix-en-Provence (2021)], which is of particular interest to physicists and chemists. Among the recurrent national events, we should also mention the User meeting of the SOLEIL synchrotron in January of each year and the Journées de la Diffusion Neutronique - Neutron Scattering Days organized by the French Neutron Scattering Association (SFN). Numerous thematic training sessions or workshops also punctuate the life of the French community and are supported, in particular, by the professional networks.

This constellation of activities and opportunities for interaction between French crystallographers reflects the diversity of this science and its great dynamism. The interface with international bodies, and in particular the IUCr and the ECA, is supported by the French Committee of International Scientific Unions (COFUSI) within the Académie des Sciences.

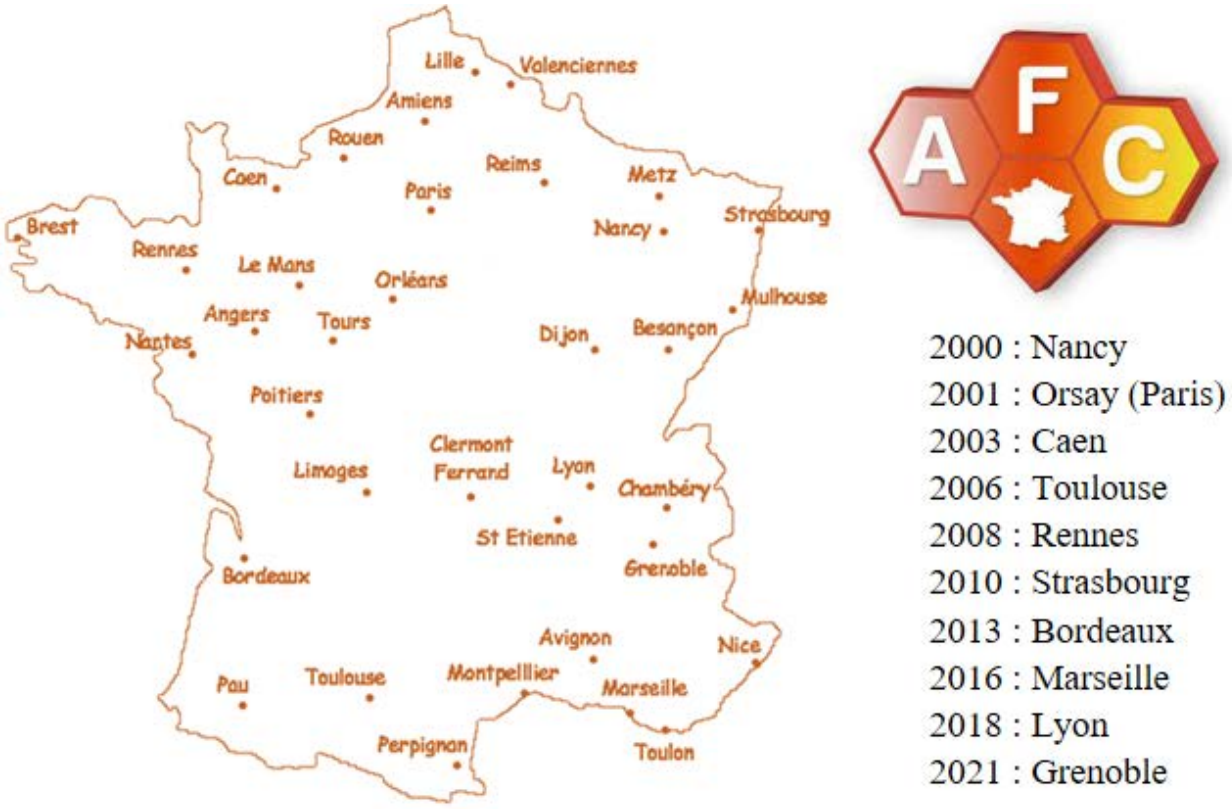


Figure 1. Map of continental France featuring notably the main cities mentioned in this article (left), the French Crystallographic Association logo (top right) and the list of AFC General Meetings held over the past two decades (right). The full list back to 1953 can be found on the Wikipedia page of the AFC.

The following paragraphs are dedicated to the recent activities covering the different branches of crystallography in France. They illustrate the complex and dense structure of crystallography in France.

2. Recent activities in biological crystallography

The French structural biology community has been very active over the recent years, standing at the forefront of many areas of biological research and contributing to key technological and methodological developments. In France, as in the rest of the world, the resolution revolution of cryo-electron microscopy (cryo-EM) combined with the development of new tools and instruments to use submicron-sized crystals for crystallographic structure determination, have allowed researchers to tackle challenging projects, most of which were simply out of reach less than a decade ago.

A major focus of recent French structural studies has been on health-related projects, and in many of the examples described below, cryo-EM and X-ray crystallography have been used hand-in-hand **to elucidate the mechanisms of action of large molecular machines and identify possible inhibitors of these complexes for therapeutic use**. Major structural biology teams from Bordeaux, Grenoble and Strasbourg have notably contributed to the structure determination of several large protein–nucleic acid complexes, including the ribosome, key complexes involved in chromatin assembly and remodelling, and also several bacterial complexes such as the RNA polymerase and DNA gyrase. French researchers from Bordeaux and Marseille have also teamed up to better understand bacterial pathogenesis by revealing, for example, the structure and mode of action of the type VI secretion system [Fig. 2(*a*)] that is used by bacteria to transfer virulence factors, such as toxins, into their cell hosts.

France hosts several expert teams in the study of **integral membrane proteins**, which are known to be very difficult to handle and poorly amenable to structural studies. To tackle these challenges, several French laboratories have contributed to the development of new expression, solubilization, stabilization, crystallization and structure determination techniques, which, together with new advances in cryo-EM imaging of membrane proteins, have allowed the elucidation of several remarkable structures. For example, in Grenoble, the Nury team has performed an in-depth study of the mouse serotonin 5-HT3

receptor in different ligand-bound conformations [Fig. 2(*b*)], and Gordeliy and coworkers have elucidated high-resolution structures of several light-sensitive receptors, including members of the rhodopsin family. These challenging structural biology projects have also benefitted from the expertise of several French theoreticians, such as Chipot and colleagues from Nancy, who bring life to these key assemblies through molecular dynamic simulations.

Structural virology is also an area in which French structural biologists from the Paris area and Grenoble are particularly well established. In recent years, Cusack and colleagues have, for instance, determined structures of several viral DNA and RNA polymerases from different classes of viruses, providing the community with new anti-viral drug targets. Investigations of the structures of entire viruses and viral nucleocapsids have also shed light on the mechanisms by which viral nucleoproteins encapsulate viral genomes. Several studies, notably those led by Gaudin and colleagues, have also focused on the critical interactions between viral glycoproteins and cellular receptors, a key step for allowing entry of viruses into the host cell. Many of these research teams are now heavily involved in COVID-19-related projects. For example, the Rey team from the Pasteur Institute in Paris, took part in some of the first structural studies of the SARS-CoV-2 spike glycoprotein [Fig. 2(*c*)] and is now actively involved in several additional COVID-19 projects.

Beyond these health-related research projects, several research teams in France are also engaged in **the development of new sustainable sources of energy and of novel means to clean-up polluted environments**, and here again, structural biology plays a key role. A consortium of researchers from Toulouse and a French biotech company, Carbios, based in Clermont-Ferrand have, for example, succeeded in engineering a "super" enzyme capable of efficiently degrading poly(ethylene terephthalate) or PET. This is the most abundant polyester plastic, largely responsible for the millions of tons of plastics that accumulate worldwide. The elucidation of the high-resolution crystal structure of this enzyme [Fig. 2(*d*)] has enabled the researchers to improve both its thermostability and catalytic properties. Other researchers from Aix Marseille University recently characterized the structure and activity of an algal enzyme capable of converting fatty acids into hydrocarbons in a single light-driven reaction. Since this initial discovery, the team has demonstrated that the use of such microbes to produce liquid transportation fuels is feasible using a simple *E. coli*-based cell factory.

These structural biology success stories would not have been possible without new **methodological and technological developments** to overcome the numerous hurdles encountered in structure determination. Here again, **many laboratories in France play major roles** in such developments. For example, a lanthanide-based molecular complex, Tb-Xo4, known as the crystallophore, has been identified by Girard and colleagues from Grenoble and Lyon. This complex facilitates crystal growth and solves the phase problem, two of the major hurdles in structural biology. Beyond the recent developments in cryo-EM mentioned above, structural biology has also recently benefited from access to new instruments, such as X-ray free-electron lasers (XFELs), and to new developments in crystallization and data collection devices, notably for serial crystallography at synchrotrons such as SOLEIL or ESRF, in order to perform time-resolved experiments on sub-micron-sized crystals. Such approaches have, for instance, provided key insights into photoswitching and photoconversion mechanisms within fluorescent proteins that occur at the nanosecond to millisecond timescale. **Serial femtosecond crystallography** has also recently been used by a large consortium of researchers to elucidate the structure of a mosquitocidal protein, Cyt1A, using submicron-sized *in vivo* grown crystals [Fig. 2(*e*)].

The French structural biology community benefits from access to a number of national and international large-scale facilities, such as SOLEIL or ESRF, and numerous technological platforms through the French Infrastructure for Integrated Structural Biology (FRISBI) centres in Strasbourg, Grenoble, Montpellier, Marseille and Paris. A French national training network in structural biology (ReNaFoBiS) was also set up in 2013 to coordinate the different teaching courses and training programmes available for students and young scientists. In 2019 and 2021, the AFC and the French Biophysics Society jointly organized the first two French biennial Integrative Structural Biology Meetings (BSI2019 and BSI2021), first in Toulouse, then in Paris-Saclay. These have been a great success, bringing together this dynamic structural biology

community for a week of scientific exchange.

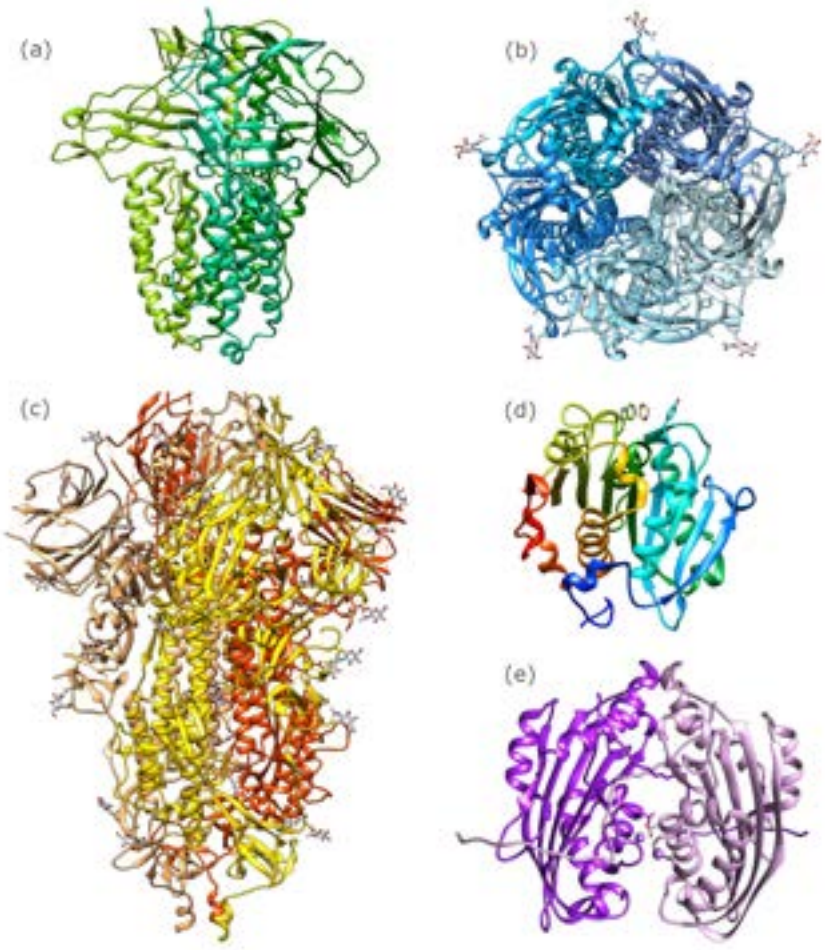


Figure 2. (*a*) Cryo-EM structure of the baseplate complex from the type VI secretion system (PDB 6GIY). (*b*) Cryo-EM structure of the pentameric mouse serotonin 5-HT3 receptor bound to palonostron (PDB 6Y1Z). (*c*) Cryo-EM structure of the trimeric SARS-CoV-2 spike protein (PDB 6VXX). (*d*) Structure of an engineered plastic-degrading (PET-hydrolase) enzyme (PDB 6THS). (*e*) Structure of the mosquitocidal Cyt1A toxin determined by serial X-ray crystallography (PDB 6T19).

3. Recent activities in chemical crystallography

Chemical crystallography is often defined as the application of diffraction techniques to the study of structural chemistry. Almost all universities and academic research centres (CNRS, CEA *etc.*) in France are equipped with X-ray diffractometers for routine and non-routine structural analyses. Most of the X-ray services are managed by only one or more trained crystallographers, and research groups dedicated to only chemical crystallography do not exist anymore in France, although many chemical crystallographers serve research groups across the whole country. Taken together, almost all aspects of chemical crystallography are covered in France.

Many chemical crystallographers are gathered in the ЯECIPROCS network, which organizes every year meetings and training sessions on a diversity of topics, very often oriented towards the mastering of new and old crystallographic software packages. The network has also implemented a national online training course on the radiation safety aspects of laboratory diffractometers.

It is difficult to specify in which subdisciplines of chemical crystallography France is particularly outstanding, but France has a very strong track record in at least four areas: development of software for chemical crystallography, high-pressure diffraction, crystallographic battery research and electron diffraction. Some recent examples of each of these areas will be given here.

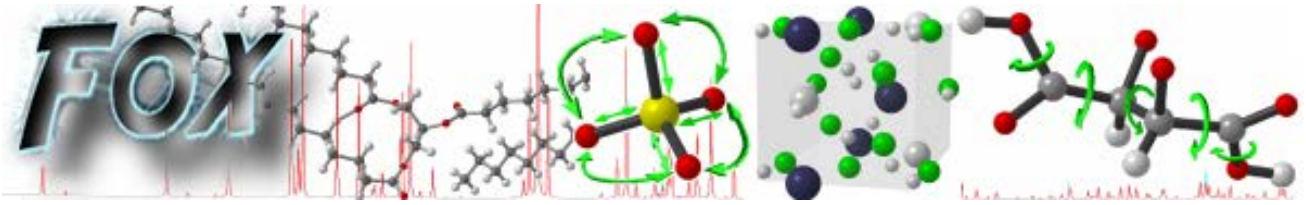


Figure 3. *FOX*, 'Free Objects for Crystallography', is the very popular and highly successful open-source program for *ab initio* structure determination from powder diffraction data.

Software for chemical crystallography

The development of software for chemical crystallography in France has been a success story for more than four decades. One of the most popular packages for Le Bail and Rietveld powder diffraction refinements, *FULLPROF*, was developed at the Institut Laue Langevin by J. Rodriguez-Carvajal in Grenoble starting from the late 1980s. This package still receives updates at least three or four times per year, implementing new functionalities and bug fixes. The Le Bail refinement part of *FULLPROF* had actually been developed before the first *FULLPROF* release by the Laboratoire des Fluorures in Le Mans. This method revolutionized the refinement of cell parameters from powder data, which was hitherto done by least-squares refinement on peak positions only but could then be performed as a full-pattern refinement without knowledge of the atomic structure. It additionally offered the possibility to obtain more accurate peak intensities for structure solution from powder data. *FULLPROF* is almost always interfaced by the *WINPLOTR* graphical software package developed since 1998 at the University of Rennes (T. Roisnel), and which is still regularly updated with new releases several times per year. Another famous program, *DICVOL*, was written at the same university, implementing the successive dichotomy method for *ab initio* peak indexing. *DICVOL* is still used all over the world as a stand-alone program or is interfaced with other programs such as *WINPLOTR*. For the intermediate step between peak indexing and structural refinements, *FOX* (Fig. 3) was developed initially at the University of Geneva before being continued after some years in Grenoble until today by V. Favre-Nicolin, co-laureate of AFC's André Guinier prize together with G. Bricogne in 2020. *FOX* is one of the most used and successful *ab initio* structure solution packages for powder diffraction. The original 2002 publication on *FOX* received more than 1300 citations, of which about 60 were in 2021. Finally, the bilateral Italian-French program *MAUD* (Caen) is still the international reference for the so-called combined approach, where data from diffraction experiments are combined with data from X-ray reflectivity or fluorescence measurements.

Several French software packages exist to analyze electron density derived from experimental single-crystal diffraction data or *ab initio* calculations. *MOPRO*, and the associated *MOPROViewer* program (University of Nancy), can be used to refine high-resolution electron densities by multipole structural refinements and also to determine and visualize derived topological quantities. *MOPRO* is also interfaced with the *ELMAM2* experimental multipolar database, which groups multipolar parameters necessary to describe the charge density of proteins and common chemical groups. Both programs are still being actively developed. Another approach for analyzing electron density properties, the non-covalent interactions (NCI) method, was made popular by the *NCIPLOT* program being coded at the Laboratory for Theoretical Chemistry in Paris (J. Contreras-Garcia). Extensions to the method, notably the Independent Gradient Model (IGM), have been proposed by researchers at the University of Reims and implemented in their *IGMPLOT* program.

Chemical high-pressure diffraction

France has several centres of excellence for chemically related high-pressure research. The group led by J. Haines at the University of Montpellier seeks to use high pressure to synthesize chemical species that cannot be synthesized otherwise. These may have very different properties at non-ambient conditions or may change drastically the properties of the materials in which they are confined at high pressure. During the last years, this ongoing research is oriented towards the high-pressure synthesis of linear or sinusoidal molecular strings inside the pores of different zeolite framework structures. The group notably tries to synthesize by high-pressure techniques confined boron nitride nanowires having exceptional photonic properties with a narrow emission for far-ultraviolet wavelengths. High-pressure diffraction, and also high-pressure Raman and infrared (IR) spectroscopy, are used to characterize the obtained materials.

A research group at Sorbonne University in Paris (S. Klotz) is specialized in high-pressure chemical research by neutron scattering, for instance, in the field of simple molecular systems under high pressure, such as solid oxygen. Another important research topic is related to the methodology and metrology of high-pressure diffraction experiments. In collaboration with Japanese groups, a new high-pressure cell for neutron diffraction experiments using nano-polycrystalline anvils has recently been proposed. This group is also involved in the International Practical Pressure Scale project, which aims to define a practical pressure scale for high-pressure research.

Special phenomena occurring in transition metal complexes such as spin-crossover transitions are thoroughly investigated in Toulouse (G. Molnár) and Bordeaux (P. Guionneau). These crossovers can be induced by pressure, temperature, light or other external fields. Molecular spin-crossover compounds can potentially be used for mechanical actuation purposes: an illuminating example was recently given for a Fe spin-crossover compound. In this case, the complete set of elastic moduli was determined by a combination of high-pressure diffraction, nuclear inelastic scattering and micromechanical measurements. Variable temperature high-pressure X-ray powder diffraction using synchrotron radiation has also revealed the actual volume variations of these materials.

Crystallographic battery research

There are several very active research groups in France that use X-ray and neutron diffraction studies to investigate the *operando* behaviour of batteries. Research groups in Amiens, Bordeaux and Paris often collaborate in the context of the French network for electrochemical energy storage (RS2E). The use very sophisticated experiments involving *operando* electrochemical cells coupled to laboratory or synchrotron diffraction or absorption spectroscopy setups. In a recent publication, they showed the feasibility of reversibly intercalating Li^+ electrochemically into VX_3 layered halides by using superconcentrated electrolytes. This was done by using a combination of electrochemical and solubility measurements with structural *operando* synchrotron X-ray and neutron diffraction. This is a change of paradigm as almost all research was until then focused on layered oxides or sulfides.

The Monconduit group at the University of Montpellier has a very strong track record in *operando* research by diffraction and pair distribution function analysis on battery systems, shifting in recent years to the study of potassium ion batteries as alternatives to relatively high-cost lithium ion batteries. The structural behaviour of systems with notably Sb and Bi electrodes has been investigated using laboratory equipment during electrochemical cycles.

Electron diffraction

During the last five years, electron diffraction for structural chemistry has been developed in France by groups in Caen and Lille. The Caen group (P. Boullay) showed in 2017, in collaboration with a group from the Institute of Physics in Prague, how hydrogen atoms could be located in organic compounds using electron diffraction data obtained from nano-sized crystals. The Roussel group in Lille is mainly involved in the structural resolution of complex natural and synthetic oxides by a combination of powder diffraction techniques and dynamic refinement of precession electron diffraction data on submicron-sized samples. Both groups are actively involved in the organization of workshops for electron diffraction in France, *e.g.* the CrystElec schools of which the third edition took place in Grenoble in October 2021. This year three different electron diffraction schools are being organized in France.

4. Recent activities in physical crystallography

Crystallography plays an important role in condensed matter physics, and the French community of crystallographers demonstrates intense activity at the forefront of various aspects of research in this domain. Below, we present just a few selected examples of the more salient recent results.

One of the most vivid parts of this activity has been for a long time the investigation of **the relationship between crystal structure and the physical properties of materials**, which was strongly boosted after the discovery of high-T_c superconducting cuprates in the late 1980s, and later on with GMR/CMR manganites or superconducting pnictides. Understanding the mechanisms at the origin of these intriguing properties has required detailed knowledge of their structural phase diagrams as a function of composition (*i.e.* electronic doping level), temperature or pressure. French crystallographers have widely contributed to such studies, with strong contributions from several research teams, notably in Orsay, Caen and Grenoble, primarily involving neutron scattering studies of crystal and magnetic structures at the Leon Brillouin Laboratory (LLB) reactor in Saclay and the Institut Laue Langevin (ILL) in Grenoble. More recently, the renewed interest for compounds displaying the coexistence/competition of different properties (*e.g.* magneto-electricity, multiferroism …) has stressed the central role of the crystal structure, as these crossed-linked effects are largely controlled by the symmetry of the material. New concepts have recently appeared in this field, such as the so-called hybrid improper-ferroelectricity, based on for example doubly ordered cation substitutions in perovskite-based compounds leading to a wealth of magnetic/structural phase transitions. Among others, the team of C. V. Colin in Grenoble has produced pioneering results to understand the physical mechanisms for multiferroism and magneto-electric behaviour of one-dimensional magnetic pyroxenes, and hybrid improper ferroelectricity and magnetism in Na*LnM*WO_6 doubly-ordered perovskites. These works, based on synchrotron and neutron diffraction measurements as a function of temperature and Rietveld refinements (using the *FULLPROF* software), make use of the symmetry-adapted analysis concept, allowing one to discriminate the distortion modes, which are relevant to the structural phase transitions. A lot of work has also been carried out by teams in Orsay (F. Damay) and Grenoble (V. Simonet) on non-conventional magnetic oxides, which display magnetic frustration and complex magnetic/structural interactions leading to anomalous magnetic orders, structural distortions or chiral effects. These results emphasize the central role played by symmetry and fine structural details for the existence of these properties, which need to be determined accurately to discriminate between various proposed mechanisms. The role of neutron scattering for these investigations must be stressed again, and the recent closure of the Orphée reactor in Saclay may represent a severe hindrance to their developments.

Phase transitions at high temperature are of paramount importance for the mechanical behaviour of industrial ceramics. A careful experimental reciprocal space investigation coupled to precise modelling of the effect of the various phenomena occurring at the transition (cracks, distortions, strain, …) on the diffraction signal has been carried out by the team of R. Guinebretière (Limoges) to reach a quantitative description of the martensitic phase transition up to 1500°C in ZrO_2 dense ceramics. This allowed one to disentangle the complex behaviour of the sample microstructure through heating and cooling and demonstrates the great potential of such experiments to understand the mechanical properties of

materials.

With the recent major advances of synchrotron sources, the interest of the community becomes more directed towards the local scale, *i.e.* ***in situ*** **and/or** ***operando*** **and fast phenomena investigations**. The boom of nanosciences forces crystallographers to adapt their experimental techniques to tackle the problems brought about by the need to describe the structure of nanomaterials at the local scale. One angle of attack is to make use of the Pair Distribution Function, obtained by Fourier transforming powder diffraction data, which provides multi-scale information on the structure and microstructure, disregarding the level of crystallinity of the material. Requiring high-energy and high-flux sources, this growing technique now benefits from well-adapted beamlines at synchrotron sources, such as the CRISTAL beamline at SOLEIL, the French-CRG beamline D2AM and several others at ESRF (ID22, ID11…). This is becoming increasingly popular in laboratories, with the availability of Ag *K*α radiation sources and efficient 2D detectors. Recent examples of applications by French teams to physics research topics stem from the *in-situ* study of catalyst nanoparticle growth, the phase transformation/amorphization of pharmaceutical molecules by high-energy milling *in situ* (Grenoble) and the study of water confinement in mesoporous materials (Nancy).

Another quite different approach to the study of local structure is the investigation of single objects using **coherent X-ray diffraction imaging**. The Aix-Marseille team of M.-I. Richard is the leader in this emerging method, which they applied, for example, to the reconstruction of catalytic single nanoparticles, allowing one to observe the strain state of the different facets and detect twin boundaries and their evolution during the catalytic process (see Fig. 4). In this context, the *PyNX* software package, developed by V. Favre-Nicolin, has rapidly become a key tool for image reconstruction from a coherent diffraction signal and ptychography. **Coherent diffraction** has also been extensively used by the Orsay/SOLEIL team of S. Ravy for the investigation of charge-density waves (CDWs), for which they have developed unique expertise. For example, they were able to provide remarkable results on the role of surface pinning on the flow of CDWs exposed to an external field. With C. Lhaulé, this team has also developed ultra-fast (subpicosecond) diffraction measurements at the CRISTAL beamline at SOLEIL, used to study out-of-equilibrium states in crystalline solids in pump–probe experiments. In Rennes, the team of E. Collet has a leading role in the study of **photo-induced phenomena by ultra-fast diffraction**. They have naturally turned to using the extraordinary possibilities provided by XFELs for this type of measurement with time resolutions down to femtoseconds. As an example, this team recently used XANES data to study the development of the coupled charge transfer between metals in a Co–Fe Prussian blue analogue and how it evolves in time, showing that the photoinduced spin transition of Co atoms from low- to high-spin state induces a charge transfer to Fe atoms from Fe^{II} to Fe^{III}. Also, using femtosecond X-ray diffraction experiments at the Swiss X-FEL, they revealed how electronic and structural precursor phenomena generate strain waves and a coherent macroscopic transformation pathway for the semiconducting-to-metal transition in bistable Ti_3O_5 nanocrystals. Such pioneering works pave the way for future discoveries in the yet unexplored domain of ultrafast physical phenomena.

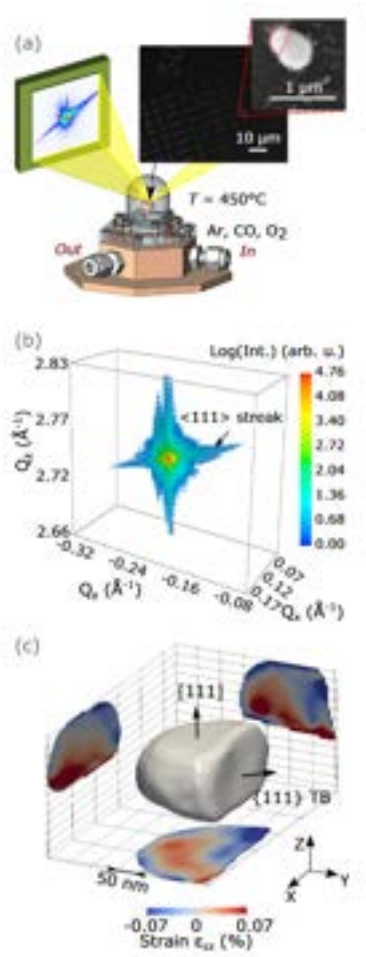


Figure 4. Experimental setup (*a*) and measured individual particle in reciprocal (*b*) and real space (*c*). J. Carnis *et al.* (2021). *Nature Commun.* **12**, 5385.

5. Recent activities in mineralogy related to crystallography

The French mineralogical community has developed strong interactions with the physical and chemical crystallography communities. Major advances have been made in fields covering a wide range of thermodynamic conditions, from ambient low-temperature conditions to the highest pressures and temperatures that can be experimentally obtained. Many of these successes have relied on combining advanced experimental studies and up-to-date molecular modelling approaches. Particular interests are devoted to the point or extended defects occurring in minerals, as well as to the changes in mineral properties when their characteristic size goes from the macroscopic scale down to nanometric particles. Departure from the ideal structure is the rule for naturally formed crystals, affecting their physical-chemical properties and bringing important information on their growth conditions and subsequent geological history.

At high pressures and temperatures, investigations aim at unravelling the nature and properties of the **materials occurring in the innermost parts of the Earth and other planets, including those orbiting around other stars**. Elucidation of the structure of the phases with relevant chemical compositions under extreme conditions is key information for the interpretation of large-scale geophysical observations, and this makes strong constraints in the establishment of geochemical balances. Concerning this last point, studies addressing the incorporation mechanisms in the **high-pressure crystal structures** of volatile elements, such as hydrogen, noble gases, halogens or oxidized forms of carbon, are particularly relevant. Such investigations are performed in Paris in close collaboration with groups specialized in high-pressure physical crystallography. Major advances at Lille and Montpellier have also been achieved in the understanding of the deformation mechanisms of major rock-forming minerals. Studies in this field include advanced electron diffraction techniques and the multi-scale modelling of deformation, from the atomic to the polycrystal scale. More generally, the study

of deformation and transformation mechanisms of minerals has important implications for understanding major geological processes ranging from convection in the Earth's mantle to the building of mountain chains. The microstructure of deformed minerals in the Earth's crust has received detailed attention because it is an indicator of stress/strain in rocks. Investigations conducted in Paris on the mechanical twinning of calcite have refined its use for paleopiezometric and tectonic studies. Geological implications of the radiation-induced transition from the crystalline to the amorphous state of radioactive minerals are also explored in Saint Etienne.

Under milder ambient conditions, a major interest is the study of the growth of crystals from aqueous solutions. Studies aiming at elucidating the **multi-step nucleation processes of sulfates, phosphates and carbonates** are conducted in Grenoble and Marseille. They have important implications for the understanding of minerals in planetary surface environments, as well as in biological fluids. Incorporation of molecular impurities in the crystal structure is of particular importance because some of these impurities are used as indicators of past conditions prevailing at the Earth's surface, contributing to paleoclimatic and paleoecologic reconstructions. Elucidating the control of crystal growth by living organisms is a major research field in biomineralogy. The formation of specific crystal shapes and their potential use as biogenicity indicators in ancient rocks has attracted detailed studies of the controlled growth of magnetites in magneto-tactic bacteria.

Also typical of hydrothermal and lower-temperature environments, clay minerals are finely divided layered silicates displaying stacking disorder, extended chemical variability and submicrometric particles. They share these properties with other natural phases with layered structures such as manganates or imogolites. A property of some of these layered minerals is also that they incorporate molecular species as well as cations in their interlayer spacing, depending on the properties of the surrounding aqueous solutions or atmosphere. Because of their ubiquitous occurrence and small grain size, they play a key role in many natural processes. The crystallography of **clay minerals** is a very active field in France with a strong relation to chemical crystallography. Studies led at Grenoble, Poitiers, Toulouse and Orsay aim at unravelling the real structure of these minerals and its relation to specific properties. In this context, development of codes dedicated to the modelling of diffraction patterns of disordered layered structures plays a key role.

Closer to crystalline perfection, but still related to crystal growth mechanisms, the natural formation of minerals of **gem quality** is a fascinating field in which researchers from Nantes and Aix-en-Provence have world-recognized expertise. Fundamental studies of the expression of crystal symmetry and twinning in minerals are being developed in Nancy. Lastly, French research activities related to mineral crystallography also benefit from the close connection to the Mineral Collections (see a great example of calcite in Fig. 5). For instance, the system of donation or loan of samples set up by the two mineral collections of Sorbonne University and of the Muséum national d'Histoire naturelle (MNHN) is very much appreciated and routinely used by the French mineralogical community.



Figure 5. Calcite from Elmwood, TN, USA. © Mineral Collection of Sorbonne University, reproduced with permission.

6. Recent activities in crystal growth

The AFC crystal growth transverse axis was set up in 2018 and is led by the French Committee for Crystal Growth (CFCC). Its two main goals are to 1) ensure national and international visibility of the French crystal growth communities and networks, both in national and in international scientific bodies such as the International Organization for Crystal Growth, the European Network of Crystal Growth, the IUCr and its Commission on Crystal Growth and Characterization of Materials; 2) organize national or international scientific events, and coordinate many initiatives (workshops, schools, national training actions, topical days *etc*.), in the realm of crystal growth and related characterizations.

Several scientific communities, technological networks and research consortia (mostly CNRS-funded) are now federated at the French level: the CRISTAL community focused on the research in industrial crystallization and precipitation; the High-Pressures network dedicated to high pressures; French members of the International Organization for Biological Crystallography (IOBCr); the CRISTECH network for crystal growth and crystallization technologies; the $CMDO^{+}$ network for optical crystals, micro- and nanostructures, and devices; the Research Group (GDR for Groupe de Recherche), PULSE, gathering the experts of basic mechanisms driving epitaxial growth, its new topics and emerging technologies, as well as the properties and applications of epitaxial systems; the GDR, SAM, dedicated to the science of solidification; the GDR, OXYFUN, that focuses on transition metal oxides in thin films and on growth and characterization of superstructures and their implementation in devices; and the RECITAL network for users of the pulsed laser deposition technique and related instrumentation applied to thin-film crystal growth.

Consequently, the research topics covered by the entire French crystal growth community **not only encompass bulk crystal growth activities but also include many more scientific and technological branches**. These include the following: fundamentals of nucleation and crystal growth

(theory, modelling and experiments designed to learn the fundamental aspects of nucleation and crystal growth); crystallization mechanisms, morphological and/or growth instabilities, growth technologies and process control; development of new methods and approaches for bulk growth, structure and properties of solid–vapour, solid–liquid and solid–solid interfaces; physical, chemical and technological aspects of thin-film formation and epitaxial growth; structural defects and impurities in crystalline materials, surface and bulk defects; crystal growth and characterization of nanostructures, low-dimensional and confined systems (nanoparticles, quantum dots, nanowires, nanotubes *etc*.); growth of advanced inorganic materials (mesocrystals and colloidal systems); crystallization in organic and biological systems (organic, macromolecular and biomolecular crystals, protein and polymer crystalline materials, current advancements in biomineralization); industrial crystallization, technologies and process control; crystal growth under a variety of external fields and extreme conditions – electric fields, magnetic fields, hyper- and microgravity, radiation, vibration, ultrasonic, high pressure, thermal and mechanical stress *etc*.; advanced growth technologies; advances in observation and characterization methods such as *in situ* monitoring methods and analysis of physical, structural and chemical properties of crystals (microscopy, spectroscopy, radiography, topography, scattering and other characterization techniques, as well as recent advances in *in situ* monitoring methods, see Fig. 6). Virtually all types of materials are addressed in the investigations covered by the AFC crystal growth transverse axis.

French teams from the crystal growth community have also contributed to several important advances in the field in recent years:

Heretofore ungrown crystals have been created, such as high-Verdet constant cubic Tb_2O_3 or highly Yb^{3+}-doped cubic Gd_2O_3 laser crystals, optical-floating zone- or flux-grown $BaZrO_3$ crystals, oxygen annealing-enhanced piezoelectric and ferroelectric flux-grown $(K,Na,Li)(Nb,Ta,Sb)O_3$ single crystals (Dr Ph. Veber - Luminescence Team/ILM, Lyon);

New *in situ* observations-based phase-field models have been implemented, including strong interfacial anisotropy effects in eutectic growth, which are now capable of accounting for crystallographic features at triple lines, opening new perspectives for epitaxial growth and nanowire growth (Dr S. Akamatsu/INP, Paris, and Professor Dr M. Plapp/Condensed Matter Physics Laboratory, Palaiseau);

New engineering strategies are being established for producing defect-free SiGe strained nanolayers and of chemical strain engineering of magnetism in oxide thin films (control of the oxygen vacancies content during the epitaxial growth of $PrVO_3$ thin films on a $SrTiO_3$ substrate);

Droplet-based microfluidic platforms have been designed for both the optimization of crystallization conditions and outputs, and the precise determination of solubilities, which is compatible with any solvent and can address an extensive range of materials, such as APIs or membrane proteins, where the coupling with UV-Vis and Raman spectroscopies also allows the study of compound polymorphism (Dr R. Grossier/CINaM, Marseille);

New devices have been developed for *i**n situ*** **and time-resolved X-ray imaging of high-melting-temperature materials** (up to 1600°C) with simultaneous recording of X-ray radiography and topography images of the solid–liquid interface. This has been applied to the solidification of silicon and superalloys. The dynamics of formation and development of structural defects (*e.g.* dislocations, twins, small-angle grain boundaries) of microstructures (dendrites) and grains can be characterized during solidification using these X-ray imaging methods (Dr N. Mangelinck-Noël, Dr G. Reinhart, Dr G. Regula/IM2NP, Marseille).

Among the most recent and significant events organized by members of the CFCC, one should mention the annual bilateral Workshop on Oxide, Dielectric and Laser single crystals (Bordeaux 2017, Idar-Oberstein 2018, Lyon 2019, Berlin 2020, Idar-Oberstein 2021, Lyon 2022), which is now alternately organized in France and Germany, and the national training actions on *X-ray diffraction applied to crystal growth* (including *in situ* techniques, Grenoble 2019) and on the *Fundamentals of crystal growth* (Lyon 2019). The CFCC is currently busy working on the organization and the academic programme of the

forthcoming 3rd European School on Crystal Growth (ESCG3, Paris, 20–23 July 2022) and on the organization of the closely related 7th European Conference on Crystal Growth (ECCG7, Paris 25–27 July 2022).

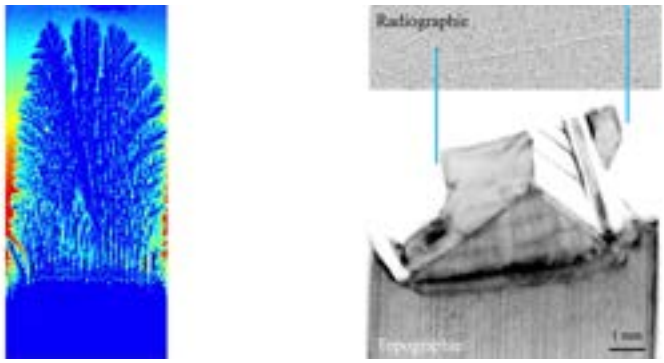


Figure 6. Left: columnar dendritic growth of an Al-Cu (4%wt) and Cu content quantification, observed *in situ* by X-ray radiography (sample width 6 mm) (courtesy of Dr G. Reinhart – MCA Team/IM2NP, Marseille). Right: solid–liquid interface, dislocations and twins in silicon observed *in situ* by X-ray radiography (top) and topography (bottom) during solidification (courtesy of Dr N. Mangelinck-Noël – MCA Team/IM2NP, Marseille).

7. Overview and perspectives on large-scale facilities in France

Experimental crystallography is fundamentally based on the interaction between condensed matter and different types of waves or particles. The development of our science is thus strongly linked to our ability to set up convenient and high-performance instruments around efficient radiation sources. In France, a large number of laboratory platforms, implemented either in individual research laboratories or increasingly shared between different research groups, are available in most of the main academic centres. Nevertheless, although very interesting technical developments are continually improving the capabilities of laboratory instruments, it is quite clear that large-scale facilities now play a crucial role in the development of crystallography. As crystallographers, we benefit in France from the implantation of both **national and European large-scale facilities** that can be classified with respect to the nature of the radiation they use. The average structures of crystals, the nature and density of defects in crystals, as well as the microstructure of polycrystals, can be studied at the quantitative level using X-rays, neutrons, electrons or NMR. Large-scale facilities allowing us to develop a wide diversity of experiments based on scattering, diffraction, absorption, emission or nuclear interaction have been implemented in France (Fig. 7).

The European Synchrotron Radiation Facility (ESRF), located in Grenoble, recently upgraded through the Extremely Bright Source (EBS) programme, is currently one of the most brilliant X-ray sources present around the world. **France is the major financial contributor to the ESRF.** In August 2020, it became the first fourth-generation synchrotron radiation source open to users. Forty-eight beamlines are currently open. Many of them are devoted to advanced experiments on crystals. Five of these beamlines are operated, as a French Collaborating Research Group (F-CRG), directly by the French scientific community and a large part of the experiments performed on these beamlines are strongly related to crystallography. The **French national synchrotron radiation source, SOLEIL,** is located near Paris. One of the main features of this source is its very broad energy range covering a large part of the electromagnetic spectrum, from infrared to hard X-rays (a few μeV to a few keV). Twenty-nine beamlines are currently open to users. Besides two beamlines devoted to structural biology, several others are dedicated to the study of single- or polycrystals through X-ray scattering, absorption or emission process. SOLEIL opened in 2008 for the users and an ambitious upgrade programme has recently been launched that will involve two phases and will end in 2030.

Besides the use of X-rays, the French crystallographers have been, for at least the past five decades, very much involved in the study of crystals using neutrons. The **Institut Laue-Langevin (ILL)** located in Grenoble near the ESRF, was inaugurated in 1971, and represents one of the most famous neutron sources around the world. It is a European source associating France, Germany and the United Kingdom

in partnership with 11 other countries. A second national reactor, Orphée, located near Paris, was operated by the Laboratoire Léon Brillouin (LLB) from 1980 until 2019. This unique situation of having two neutron reactors in France has favoured the growth of a large French neutron users' community that brings together roughly 2000 researchers, of which a large part are crystallographers. This community, led by the Fédération Française de Diffusion Neutronique, is in charge of six different instruments located at the ILL and is heavily involved in the development of new instruments for the European Spallation Source (ESS) in Lund, Sweden. Moreover, the project of a national, medium-scale neutron source has also been launched.

At the interface between material science and crystallography, the community of French electron microscopists has built a vast network that brings together the **larger French electron microscopy centres and the atom probe tomography facilities**. This network, called **METSA** (for Transmission Electron Microscopy and Atom Probe), associating eight regional platforms, offers a large number of unique up-to-date instruments. Similarly, NMR instruments spread over seven scientific high-level platforms are organized through the IR-RMN network. Mainly devoted to the study of solid-state NMR, this network is largely used by crystallographers. Access to the facilities within these two networks is organized through periodic calls for proposals as for access to large-scale X-ray and neutron sources.



Figure 7. Aerial view of the large-scale facilities located in France and presented in this article: ESRF, ILL and SOLEIL.

8. A few words on teaching crystallography

Teaching crystallography is a fascinating and difficult task. This is not a French specificity, of course, but a common, universal trait, which originates in the eminently transdisciplinary character of our science. In France, as in many other countries, the teaching of crystallography is being cut back as university curricula are revised. The erosion is rapid as in France the content of university courses is discussed and redefined every four or five years. The erosion seems to be definitive as the general trend is to reduce the volume of teaching hours. In this context, defending a teaching module explicitly bearing the term "crystallography" is a Herculean task. However, whether in chemistry, biology, geology or physics, knowledge of crystallography constitutes the pillar, albeit sometimes masked, of many courses in Bachelor's (3 years) and Master's (5 years) degrees in materials science, solid-state physics and chemistry, biophysics or archaeometry for example. Whereas the standardization of degrees in Europe (LMD system) has had the consequence of restricting degrees focusing on crystallography, it has made it possible to teach crystallography at the doctoral level. A large number of courses for students in the course of their theses are offered on university campuses in France. This may be considered a little late, but it has the merit of providing a high level of specialized training centred on concrete problems such as the refinement of crystal structures of macromolecules, the determination of microstructural parameters or integrative structural determination in biology. To overcome the lack of initial training in crystallography, many ingenious initiatives are being undertaken, for example in the form of Massive Open Online Courses or MOOCs.

Also, the dichotomy between the remarkable dynamism of the crystallographic community in France and the steady loss of substantial educational courses in this discipline is starting to move the lines. The 2014 International Year of Crystallography driven by the IUCr was no stranger to this. In this year, indeed, France was the scene of an incredible number of events of all kinds in tribute to crystallography. **The enthusiasm of the general public** gave a new energy to all the French actors of crystallography and resonated in many minds. A first major repercussion of this event was the reintroduction in 2019 of crystallography in the national high-school science curriculum. Marking a major comeback for younger students, crystallography has proved difficult to handle for existing secondary school teachers, most of whom come from a background lacking crystallography. They have then to be trained. **Many initiatives** in this direction are underway, thanks, for example, to the *Maisons pour la Science* (Science Houses) present on most university campuses, which enable French secondary school teachers to meet with academics. At this stage, let's hope that this whole chain will be successfully implemented and will allow continuity of teaching from high-school years to PhD years, and beyond.

For the younger audience or the general public, France has also built on the dynamism instilled in 2014. Each year, the AFC supports scientific mediation events around crystallography. Among them, **the crystal growth competitions** that are organised annually in many university centres (Bordeaux, Nancy, Marseille, Strasbourg ...) reach several thousands of secondary school pupils and allow many educationalists to exchange knowledge about crystallography. **All this is positive**; the teaching of crystallography seems to be recovering slightly. However, in France as elsewhere, maintaining a continuous, non-optional teaching of crystallography over a long part of the higher education curriculum probably borders on the myth of Sisyphus.

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MEETING REPORT (IUCR SUPPORTED)

To.Sc.Al'And: Total Scattering for Nanotechnology in Al'Andalus



Norberto Masciocchi, Antonietta Guagliardi, Jorge A. R. Navarro

During the last decade, staff researchers at To.Sca.Lab (Total Scattering Laboratory, http://toscalab.uninsubria.it), the joint laboratory merging experimental and computational aspects of scattering techniques (co-founded by the University of Insubria and the Institute of Crystallography of the Italian National Research Council), have organized several International Summer Schools, including the many editions of To.Sca.Lake (*Total Scattering for Nanotechnology on the Como Lake*, 2015, 2017, 2019), and successfully exported the format to Latin America (*To.Sca.Lat.*, 2018 edition, held in Florianópolis, Brazil). With the goal of widening the audience of these new approaches to the structural and microstructural characterization of nanosized materials, the 2021 edition of the Workshop, entitled *To.Sc.Al'And*, was organized as an *in-person* meeting at the Faculty of Sciences of the University of Granada (Spain), with a specific focus on topics of interest to an international gathering of young researchers, post-docs, PhD and undergraduate students. This was indeed possible thanks to the logistical support of the University of Granada and the concomitant health situation, which, during summer 2021, saw a rapid decline of the COVID-19 pandemic, particularly in the south of Spain (Andalusia).

The Workshop lasted five days (20–24 September 2021) and included lessons, demos and *hands-on* tutorials. It was attended by more than 55 young researchers, internationally renowned teachers and commercial sponsors, all participating in a very active manner in the different sessions. The wide international attendance, with students from 13 different countries, provided a rich cultural and scientific exchange environment.

The teachers were Federica Bertolotti, Simon Billinge, Hans-Beat Bürgi, Antonio Cervellino, José Manuel Delgado-Lopez, Fabio Ferri, Tatiana Gorelik, Antonella Guagliardi, Daniel Maspoch, Norberto Masciocchi, Jan Skov Pedersen and Nora Ventosa, who presented scattering techniques at the fundamental levels, including both theoretical and experimental aspects [small-angle X-ray scattering, wide-angle total scattering in direct (PDF) and reciprocal (DSE) spaces, 3D-PDF, static and dynamic light scattering and advanced applications for a wide spectrum of (organic/inorganic/hybrid/biomimetic) materials]. Hands-on sessions on data analysis with *PDFGui* and *DebUsSy* programs, developed in recent years by some of the speakers, a (live-commented) video-recorded experiment on DLS measurement on colloidal quantum dots and an additional session dedicated to oral contributions by more than 10 young participants, were also organized and duly attended.

Organizing this Workshop was possible thanks to the funds raised from registration fees, sponsorship from national and international scientific bodies (IUCr, ICDD, AIC, ECA and INSTM) and the generous

contribution by commercial sponsors, without which the organization of this Workshop would not have been possible. All such funds were indeed used to support the participation of more than 20 young and distant attendees, in the form of reimbursement of traveling and accommodation expenses or waiving registration fees. As a final comment, the organizers found it very rewarding that, having gathered the students, teachers and sponsors together in Granada, all the attendees committed themselves fully to the realization of this *in-person* event, awaited for several months after the outbreak of the COVID-19 pandemic.

NOTICE

CCDC 2022 Virtual Workshops



The Cambridge Crystallographic Data Centre (CCDC) will be hosting the following Virtual Workshops in May 2022:

Searching crystal structure data in the CSD with *ConQuest* and *WebCSD*

The basics of *ConQuest* searching

Use of some advanced query options

Introduction to *WebCSD*

The differences between searching with *ConQuest* and with *WebCSD*

Date: 4 May 2022

Time: 15:00 to 16:45 (GMT)

Register here

How to design solid structures and assess their stability in *Mercury*

Overview of the tools available in the CSD-Materials suite, focusing on tools for:

data-driven solid form design

solid form assessment during early stages of the development process

How to use *Mercury* to perform:

Crystal Packing Feature searches

Crystal Packing Similarity searches

Two identical sessions will be run on two dates to cover more time zones:

Session 1

Date: 10 May 2022

Time: 09:30 to 11:15 (BST)

Register here

Session 2

Date: 12 May 2022

Time: 21:30 to 23:45 (BST)

Register here

First steps in protein-ligand docking with *GOLD*

The basics of the Hermes interface, the CCDC's 3D visualiser for proteins

Step-by-step basics of *GOLD*

Where to get started with your docking simulation and how to run a standard protein-ligand dock with *GOLD*

How to identify the correct binding modes reliably and with confidence

Date: 18 May 2022

Time: 11:00 to 12:45 (BST)

Register here

All are free to attend. Other forthcoming CCDC events can be viewed here.



FEATURE ARTICLE

Ágnes Csanády's flowers: quasicrystals



Istvan Hargittai, Magdolna Hargittai

The date of the discovery of quasicrystals by Dan Shechtman may be assigned 12 November 1984 when the publication reporting it appeared (Shechtman *et al*., 1984 [1]). However, for science history, it is to be remembered that it was 40 years ago, on 8 April 1982, that Shechtman noted in his lab journal his observation of the electron diffraction pattern no. 1725: “10 Fold ???” (a sample of aluminum and 25% manganese) after he counted the bright spots, equally spaced from the center and from each other in the diffraction pattern. He uttered these words: “There is no such animal” (Hargittai, 2011 [2]). He knew that according to the then rules of crystallography no fivefold — or its multiples — symmetry was allowed in extended structures. It took Shechtman two and a half years to have his observation published, a few more years to have his discovery accepted by the broader community of chemists, physicists, crystallographers and materials scientists, 18 years to be awarded the most prestigious Aminoff Prize, and 29 years to be awarded the Nobel Prize in Chemistry {the pre-Nobel saga of the quasicrystal discovery is discussed in Hargittai & Hargittai (2000) [3]}.



Flower-like Al_4Mn quasicrystals decorated the cover of the October 1997 issue of *The Chemical Intelligencer*. Ágnes Csanády (Budapest) prepared the quasicrystals by rapid cooling and isolating them for scanning pictures of an $Al_{17}Mn$ sample. Hans-Ude Nissen (Zurich) took the scanning electron micrograph (Nissen, Csanády *et al.* 1988 [4]). Photograph courtesy of Ágnes Csanády.

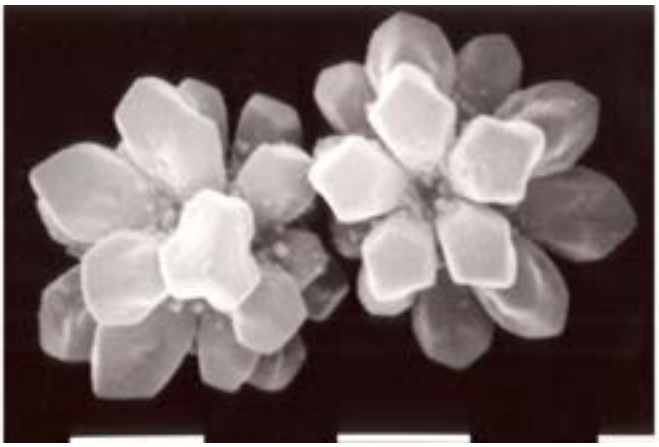
The appearance of beautiful, often flower-like, images of quasicrystals helped to keep the attention and interest in the discovery alive in the lean years. This was while Shechtman demonstrated perseverance in maintaining that his observation should not be misinterpreted by the presence of twin crystals or in any other way. Ágnes Csanády (*née* Bodoky) was an unsung hero for producing the world's most beautiful quasicrystals in the laboratory. She graduated as a Dipl.-Chemist (master's degree equivalent) in 1958 from Eötvös University Budapest. She earned her PhD-equivalent doctorate in 1983 and her higher doctorate, DSc, in 1998. She held a variety of jobs in condensed-state materials science and technology, with electron microscopy as the principal research tool. Her most productive period was 1971–1997, including the time from the mid-1980s when she became much involved with quasicrystals. She prepared them by Al–Mn and Al–Fe thin-film reactions through *in situ* treatments, not only by quenching. She did this, in part, working with a high-voltage electron microscope at the Max Planck Institute for Physics in Büsnau, Germany (Csanády, Urban, Mayer & Barna, 1986 [5]). Her principal position was at the Research Institute of Non-Ferrous Metal Industry in Budapest. This institution eventually incorporated the Hungarian research and development agency of the aluminum industry. She held visiting positions in Büsnau and at the ETH Zurich. The images of her quasicrystals made the covers of various publications; an example is shown above.

One of the papers of which both Csanády and Hans-Ude Nissen were co-authors (Nissen, Csanády *et al.*, 1988 [4]) communicated the results of scanning electron microscopy (SEM) and transmission electron microscopy (TEM) of Al_6Mn and $Al_{17}Mn$ alloys. Nissen was a section chief at the Laboratory of Solid State Physics, Eidgenössische Technische Hochschule, ETH (Swiss Federal Institute of Technology), Zurich. For Al_6Mn, the pentagonal dodecahedron form dominated. For $Al_{17}Mn$, flower-like quasicrystal units with icosahedral point symmetry appeared to be the basic forms. These flower-like quasicrystals were simply referred to as "flowers," and they could be described by a star polyhedron model based on a {5,6} Archimedean polyhedron.

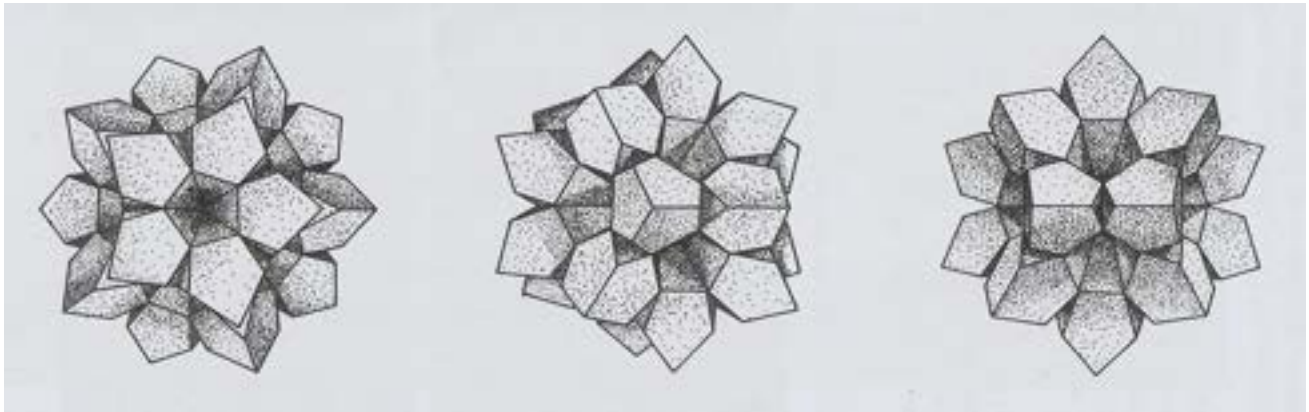


Ágnes Csanády and Dan Shechtman in 1995 in Balatonfüred, Hungary, during an international school on quasicrystals (left, photograph by I. Hargittai) and in 2019 at the Budapest University of Technology and Economics, following Shechtman's inaugural lecture as John von Neumann Professor (right, photograph by an unknown photographer).

Shechtman always appreciated Csanády's "art." Two images recall their meetings. In May 1995, one of us (IH), along with others, organized an international school/conference on quasicrystals in a resort place at Lake Balaton, Hungary. There, we could learn and discuss everything known by then about quasicrystals in an unhurried and relaxed atmosphere. On this occasion, Csanády personally demonstrated to Shechtman and everybody else her unrivalled quasicrystal images.



Scanning electron microscopy image of flower-like quasicrystals isolated from quenched $Al_{17}Mn$ ribbons. Courtesy of Ágnes Csanády (1987 [6]). The length of the horizontal bars corresponds to 1 µm.



From left to right: fivefold, threefold and twofold projection models that could also be observed in the SEM micrographs (Nissen, Csanády *et al.* 1988 [4]).

Nissen, Csanády *et al.* (1988) derived the polyhedral star morphology of the flower-like quasicrystals from one of the Archimedean (semiregular) polyhedra, *viz*. the truncated icosahedron. With this notion of icosahedral symmetry, they beautifully connected two great contemporary discoveries in materials science: quasicrystals and buckminsterfullerene.



Israeli postage stamp 2014.

One of Csanády's flower-like icosahedral quasicrystal images (Csanády, 1987 [6]) served as a basis for a beautiful Israeli postage stamp. In the introductory paragraph, we alluded to the difficulties in recognition of Shechtman's discovery. His Nobel Prize in 2011 in Chemistry ended any hesitation for such recognition. By the time the International Year of Crystallography came about in 2014, it was considered to be one of the stellar achievements of recent science. The Israeli Post Office honored both Shechtman's accomplishment and the International Year of Crystallography with its stamp. Csanády's quasicrystal provided a scientifically sound and visually pleasing artwork, attractive for scientists and philatelists alike. It turned out to be a profound tool for science popularization as well.

We thank Ágnes Csanády and Danny Shechtman for comments.

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FEATURE ARTICLE

The mystery of the Lady of the Lamp



Mike Glazer

In 1976, I moved from Cambridge to Oxford, taking all the records, crystal models and equipment of the Cambridge Crystallography Laboratory. This had originally been set up when W. L. Bragg moved from Manchester to Cambridge in 1937. I joined the lab in 1969 to work under Helen Megaw, and the head of the laboratory was William Hodge Taylor (WHT). Bragg had departed from Cambridge in 1953 to go to the Royal Institution in London.

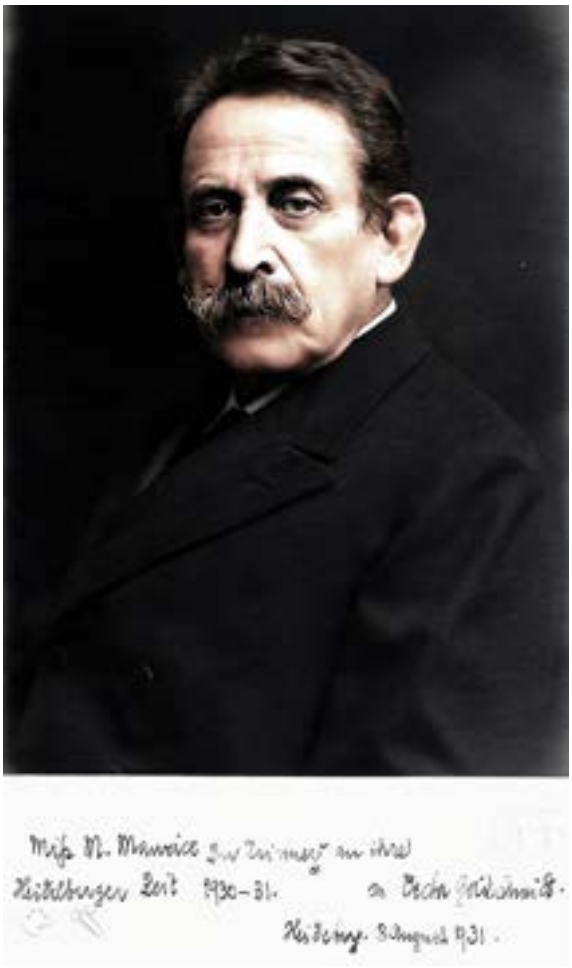


Figure 1. Victor Mordechai Goldschmidt (image colourised by AMG).

Now, one of the items (Fig. 1) that I brought with me is a photograph of the mineralogist/crystallographer Victor Mordechai Goldschmidt (1853–1933) [1]. At the bottom of the photo is an inscription written in German, saying "Miss M. Maurice in memory of your time in Heidelberg 1930–31". It is signed and dated Heidelberg, 8 August 1931. I have been wondering for a long time about this photo. Why was it in the Cavendish Laboratory, and whose office did it come from? My guess is that it came from Helen Megaw, but it could have been from WHT, who had kept items from Bragg's office after he left Cambridge, or possibly from Bragg himself. And who was Miss Maurice? What was her connection to the Crystallography Laboratory?

I eventually discovered that she was Monica Maurice (see her Wikipedia entry: https://en.wikipedia.org/wiki/Monica_Maurice), see Fig. 2.



Figure 2. Monica Maurice (copyright the Institute of Engineering and Technology and the Women's Engineering Society, colourised by AMG).

Now, she was a really fascinating woman, an aerobatic pilot (a friend of Amy Johnson, for instance), a racing car enthusiast, and obviously very talented and unusual for her time. She was also well known as an engineer. As a pilot myself, I would have loved to have been able to meet her and gossip about aircraft. When Monica was married, she chose a bright red wedding dress which caused quite a stir and even made the national news: the dress is now at the Victoria and Albert Museum in London. According to her Wikipedia entry: *In 1931, she was apprenticed as an engineer in Germany with the former parent company, Friemann and Wolf, then the world's largest and most celebrated firm of mine lamp makers. She trained in the drawing office, laboratory, machine shops and foundry, among other departments. For practical experience, she also visited the coal mines of Westphalia. She made several other visits to Germany throughout the 1930s, and on one occasion, she wandered into a restricted zone and saw what she thought was a guidance system. This she reported to the British authorities on her return, but they were not interested. At the outbreak of war, though, she was summoned to London for a three-day debriefing. All this experience, her knowledge of technical German, and her familiarity with*

many of the industrial sites, proved invaluable. Monica Maurice was known in Yorkshire as the "Lady of the Lamp" to the coal miners (see Fig. 3). The company where she was a managing director, the Wolf Safety Lamp Co. Ltd, still exists today (https://www.wolfsafety.com/history). I am grateful to Helen Close of the Women's Engineering Sociey for a link to an interesting video in which Monica appears in old age: see https://www.youtube.com/watch?v=SRMPy2BSEP4.



Figure 3. Monica Maurice, the Lady of the Lamp, in 1939 (photograph by permission of her son, John Jackson; image colourised by AMG).

In 2013, when we were planning the Two Braggs Exhibition at Warwick University (www.amg122.com/twobraggs/), I received a photograph from Robin Marshall in Manchester who was researching the history of the Physics Department. The photograph (Fig. 4), which he eventually published in his book *Three Centuries of Manchester Physics: Volume 3 1907-1937*, is of a department outing to the Blue John mine near Castleton in Derbyshire in 1929. Blue John is a particularly rare variety of the mineral fluorite, CaF_2. I can strongly recommend a visit to the mine if you ever have a chance. The photo was probably taken by Bragg himself, as his wife is there in the group but he is not. It shows the members and visitors of Bragg's group in which everyone is named. Many famous names will be well known to crystallographers of a certain age. To my surprise, I found among a group of women, including Lady Bragg, the name M. Maurice! So clearly, she had some connection with Bragg, though it is not clear what that connection was. It may be because of her association with mines, but the earlier link to Goldschmidt suggests to me that it was something else. Fig. 5 shows the site today.



Figure 4. W. L. Bragg's group on a day out to the Blue John mine in 1929. Left to right: W. Jackson, R. W. James, W. Zachariasen, B. E. Warren, R. G. Wood, H. Bell, W. Kay, E. C. S. Dickson, W. H. Taylor (obscured), J. M. Nuttall, F. Fothergill, H. Buckley, M. Jones, A. A. Newbold, J. W. Cuthbertson. E. G. Jones, P. Jones, M. Maurice, Mrs Brentano, Mrs Bragg, Dr Ito, Mrs Bradley, A. J. Bradley, Dr Naray-Szabo, A. L. Roussin (image colourised by AMG).



Figure 5. Entrance to the Blue John mine today.

When I was at the Cavendish, I recall being told by someone that Helen Megaw had been part of a team of scientists who had been sent to Germany just after World War II. She believed that she was sent there to visit the scientific research laboratories to help the German scientists recover from the terrible events of the war. But, I was also told that she had not realised that the mission was actually more about appropriating valuable equipment and knowledge to bring back to Britain. I think she would not have approved. Indeed, I have in my possession a beautiful German balance which I was told came from that time. This is where things become interesting. After the war in 1945, an organization known as BIOS (British Intelligence Objectives Sub-Committee) was established by the UK government under the chairmanship of the chemist R. P. Linstead (1902–1966) for the purpose of coordinating British scientific and technical exploitation efforts. According to Charlie Hall in his book *British Exploitation of German Science and Technology*, *1943-1949*, published by Routledge, Monica Maurice was part of a BIOS mission to Germany in April–May 1947. So I can guess that Helen Megaw was also a member of that same BIOS team, and it is then likely that they would have met. I can well imagine that Monica Maurice, knowing that Helen was a crystallographer and mineralogist (and particularly well known at the time for her work on the structure of the perovskite, barium titanate), might have given Helen the Goldschmidt photograph. Alternatively, Monica may have given the photograph on another occasion directly to Bragg, for we have seen that she was already in touch with him many years earlier.

So there the mystery remains. I would love to know the true connection between Monica and the crystallographers. If anyone out there can add to this story, please let me know.

Note

[1] Victor Mordechai Goldschmidt (1853–1933) is especially known today for the *Goldschmidt Tolerance Factor,* which uses ionic radii to establish stability of perovskites.

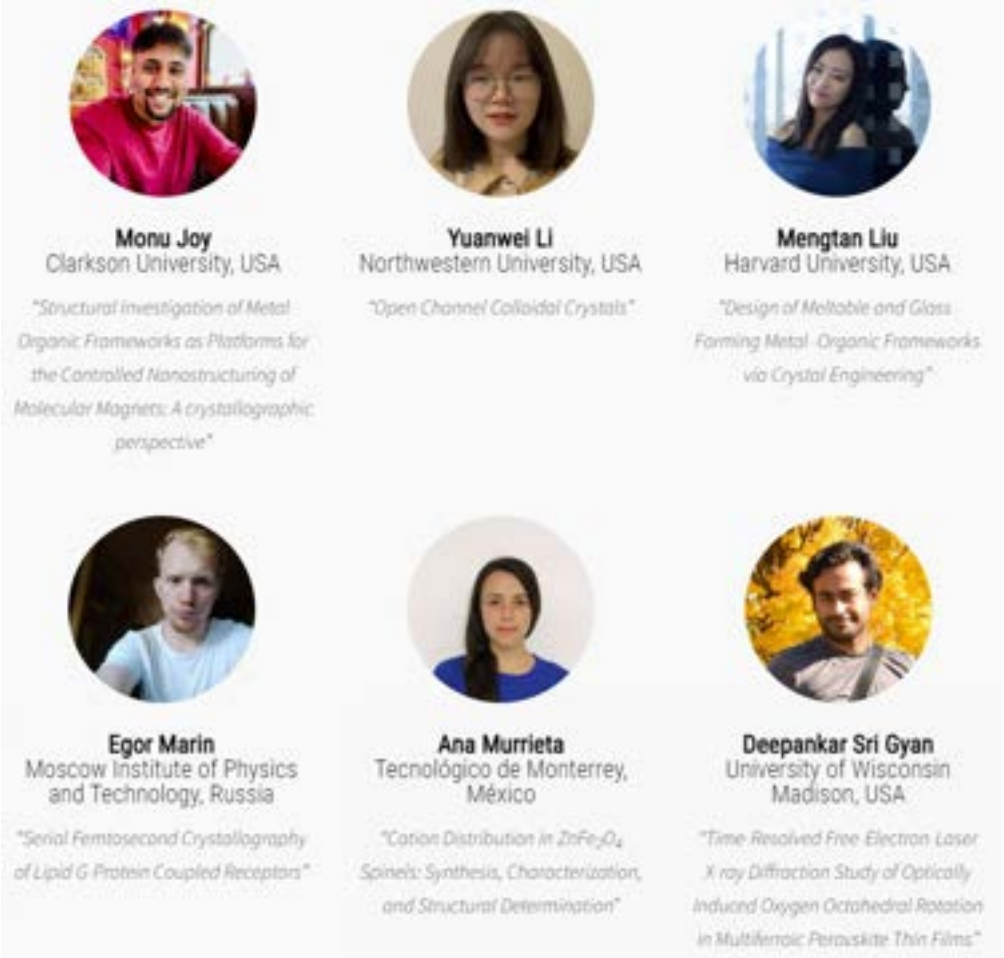
AWARDS AND PRIZES

2022 Ludo Frevel Crystallography Scholarship Awards



To encourage promising graduate students to pursue crystallographically oriented research, the International Centre for Diffraction Data (ICDD) established a Crystallography Scholarship Fund, known as the Ludo Frevel Crystallography Scholarship Fund, in 1991.

The ICDD Ludo Frevel Crystallography Scholarship Committee has selected six recipients for the 2022 Scholarship Program. These recipients were selected on a competitive basis, from 50 applications received by the ICDD Scholarship Committee.

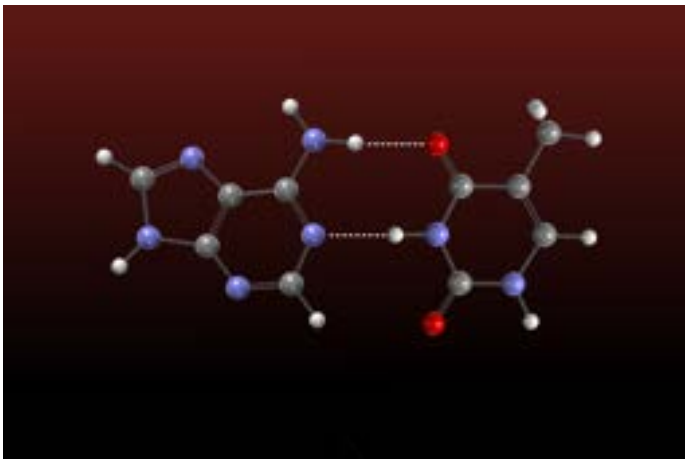


The ICDD will present each of these students with a check in the amount of USD2500 to assist in the continuation of studies in their selected fields of crystallographic research.

Applications for the 2023 Scholarship Awards open in August 2022 at https://www.icdd.com/ludo-frevel-scholarship/.

OBITUARY

June Lindsey (née Broomhead; 1922–2021)



Jane Lindsey, Robin Lindsey, Alex MacKenzie

June Lindsey, the crystallographer who determined the crystal structure of the purines, died of old age on 4 November 2021 in Ottawa, Canada, at the age of 99. The daughter of a mining engineer, her impressive scientific trajectory was by no means preordained. Born in a small Yorkshire village in England, June attended the local grammar school for girls. The renowned physicist Ernest Rutherford died in 1937, and June, then 15, was taken by the accounts of his revolutionary work on the atom. Reading Rutherford's biography only impressed her further and introduced her to Cambridge's storied Cavendish Laboratory, where he'd done much of his work. She resolved to do all she could to attend Cambridge. This involved overcoming barriers, including proficiency in Latin, a topic not taught at the girls' school. Luckily, one of the Latin teachers at the boys' grammar school offered to tutor her. Her determination and ability paid off, and she was accepted to Cambridge on a full scholarship, the first from her school to attend the University.



June Lindsey (née Broomhead) at her graduation.

Coming from the sixth form, June had not yet chosen her scientific specialty, but once at Cambridge, soon became aware of Lawrence Bragg and the crystallographic work at the Cavendish. As she later wrote, "When I first went up to Cambridge, I hadn't even heard of [X-ray crystallography], but by a fortunate fluke, chose to study just those sciences which are most essential. These were physics, chemistry, mineralogy, and mathematics." Given the field required "great patience and quite a lot of intuition", she was was naturally drawn to it. As she would later state, "One collects one's experimental data ... and then retires to the desk for many hours of thinking and calculating". However, in 1944, upon graduation (as a woman, she had to wait over 50 years for the University to grant her a BA retrospectively), she was initially compelled to teach physics at a girls' school until the Second World War ended. She had been awarded a research studentship at graduation, so in 1946 she returned enthusiastically to the Cavendish to pursue a PhD under W. H. (Will) Taylor. Once there, June developed a close friendship with Edinburgh-trained physicist Bill Cochran, to whom she attributed much of her success.

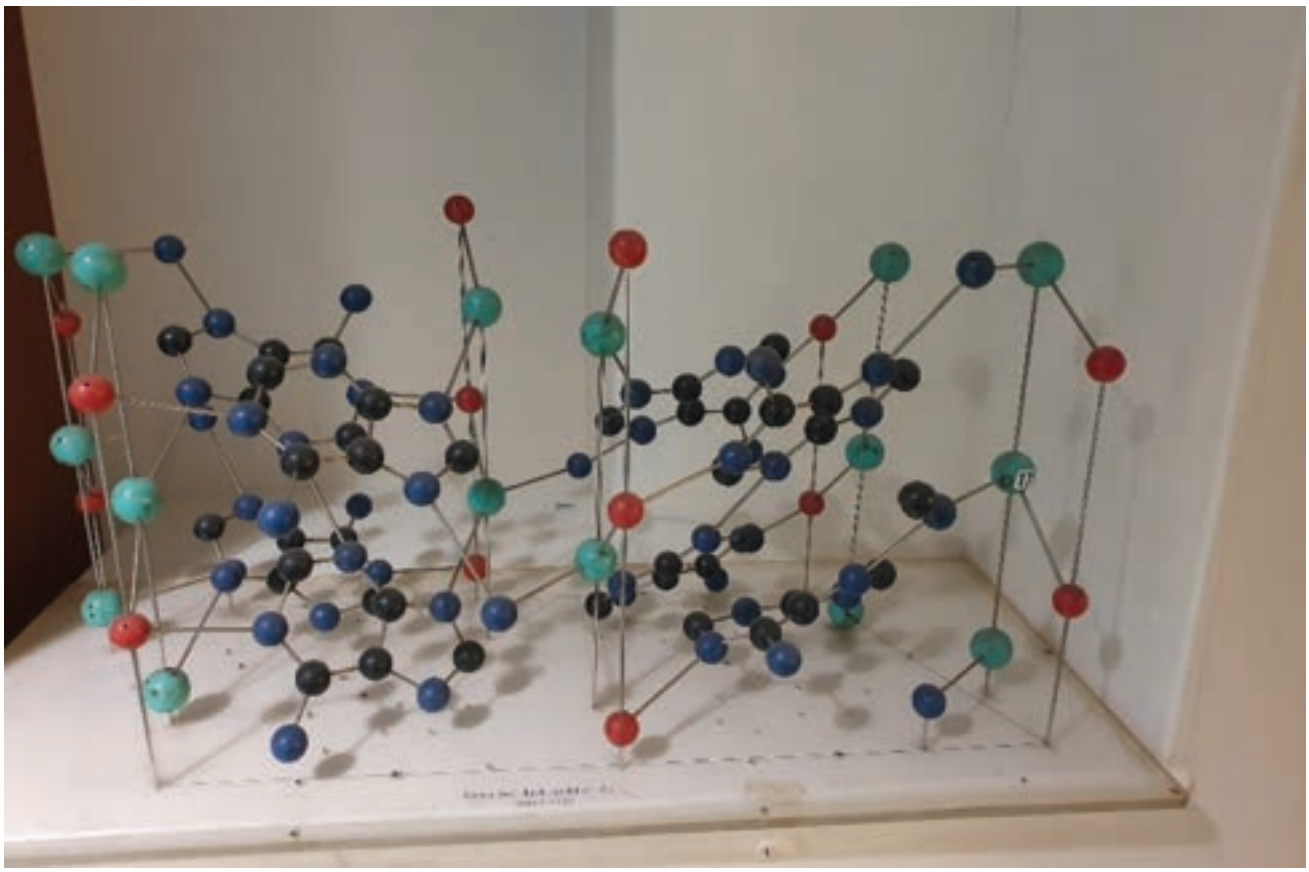


June Lindsey and Bill Cochran.

In August 1948, she submitted "The structure of pyrimidines and purines. II. A determination of the structure of adenine hydrochloride by X-ray methods" to the new journal *Acta Crystallographica*, where it was published in the first volume (Broomhead, 1948). In addition to measuring the physical dimensions of adenine, she stated, "Hydrogen bonds linking a purine molecule to chlorine atoms and to water molecules, and short van der Waals contacts with other purine molecules lie approximately in the plane of the molecule." This seemingly trivial observation triggered a critical epiphany for James Watson six years later as he worked out how the two strands of the linear nucleic acids could be bound to one another in DNA. As he writes in *The Double Helix*, "I was drawing the fused rings of adenine on paper. Suddenly I realized the profound implications of a DNA structure in which the adenine residue formed hydrogen bonds similar to those found in crystals of pure adenine" (Watson, 1968).

Over the next three days, using models of the nucleotides based on Lindsey's dimensions, Watson tried to join the nucleic acid strands, lining adenine up with adenine. Encountering difficulty, he then realized that an adenine hydrogen bonded to a thymine was the same size as the other two constituents of DNA,

cytosine and guanine, the structure of which Lindsey had also solved (Broomhead, 1951), hydrogen-bonded with one another. The two nucleic acid strands could thus be evenly joined, adenines linked with thymines and cytosines with guanines, while satisfying Erwin Chargaff's earlier observation that DNA comprised equal quantities of adenines and thymines as well as of cytosines and guanines (Chargaff, 1950).



June's 1948 model of the structure of adenine hydrochloride (A. M. Glazer's crystallography collection).

Following her doctorate, Lindsey moved to Oxford to work with Dorothy Hodgkin on vitamin B12 (Brink *et al.*, 1954). This was another rewarding experience, although frustrating at times, because after spending months on laborious calculations, Hodgkin would look over her shoulder and quickly make key observations on the structure. In 1951, she moved to Ottawa with her new Canadian husband, George, who had also earned a PhD in physics at Cambridge. She took a postdoctoral position at the National Research Council, and working once again in solitude, identified the crystal structure of codeine (Lindsey & Barnes, 1955). It was not to last, however, when she moved with her husband to Montréal and following the birth of two children, she retired after almost a decade of impressive and profoundly consequential research.

Lindsey's essential worth as a scientist was captured in a heartfelt letter Bragg wrote to her in 1952, decrying the inherent complexity of the hemoglobin structure that Perutz *et al.* were working on and how very suited she would have been for the problem. He closes the letter with the words, "We badly need your hands to tackle knotty crystallographic problems, both experimental and theoretical. I wish all these things had come up while you were still with us; they would have been just in your line."

June was a woman of many parts, despite being a rational scientist. Like her spiritualist grandmother, she had her own ghost stories, which she admitted had no rational explanation. The accountant poltergeist who broke plates in their Yorkshire home and then, with a guilty conscience, carefully swept them into a neat pile. Her spiritualist grandmother, having promised June she would visit in Canada, one day shouted to June her arrival from inside the house. When June ran down to check, no one was there, and a telegram came later that day, announcing that her grandmother had died three days before.

In many ways, June was characteristic of her era in terms of her worldview. She downplayed any gender discrimination that she experienced, whether being excluded from the Cambridge pub where the men repaired to discuss their work or having to check in and out using time cards while crystallizing codeine in Ottawa. And she would occasionally assert men's superior scientific acumen, much to her daughter's chagrin, who wonders whether she would have felt the same and made the same choices had she been born 50 years later.

A family friend captured June's personality perfectly. "She was unique in terms of her friendliness, curiosity, and in speaking her mind. Her unassuming nature and modesty tended to hide her formidable intellect and accomplishments. She did not suffer fools gladly but would listen to a well-constructed argument before concluding the debate with a devastating one-liner."

June was many things to many people: to her children, she was an old-fashioned English mother; to her friends, she was loyal and funny; and to science, a gifted researcher whose work was central to the discovery of the double helix.

June was predeceased by her husband, George, in 2011. She leaves her daughter, Jane, a senior research scientist at Harvard University's T.H. Chan School of Public Health; her son, Robin, an economics professor at the Sauder School of Business at the University of British Columbia; her daughter-in-law, Laura; and a granddaughter, Amanda.

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Editor's note: readers fascinated by the DNA story may be interested in a historical fiction account of Rosalind Franklin by Marie Benedict: *Her Hidden Genius*. Read a review by Katie Langin in *Science* here.

OBITUARY

Jack David Dunitz (1923–2021) and chemical crystallography



Jack on the occasion of his 80th birthday (photo taken by Jay Siegel).

Hans-Beat Bürgi

Jack Dunitz, emeritus professor of chemical crystallography at the Organic Chemistry Laboratory of Eidgenössische Technische Hochschule Zürich (ETHZ), passed away on 12 September 2021 at the age of 98. Jack was a leading figure in chemical crystallography. He addressed and solved a broad range of chemical problems with the help of structural information from diffraction experiments.

Attempting to appreciate Jack's merits in science runs the risk of bias. This danger can be minimized, however, by listening to Jack's own words. In an essay entitled 'La Primavera' (Dunitz, 2013), Jack reflects on his formative years, the time before he took his position at ETHZ in 1957; in 2021 Jack looked back on his long life in science in two interviews[1] conducted by E. Carreira, his colleague at ETHZ and editor-in-chief of the *Journal of the American Chemical Society*; Jack's relationship vis-à-vis molecular biology is the theme of a conversation with E. Carafoli conducted in 2017 (Carafoli, 2017); in the journal *Chimia* Jack chats about exploring the world of science, his wanderings and his life in Zürich, inside and outside of ETHZ (Weber, 2011). This account attempts to portray Jack's most important achievements against the background of concepts and activities prevailing during the time of his involvement in science. Hargittai (2021) writes in a similar vein.

Jack was born on 29 March 1923 in Glasgow (Scotland) where he grew up into the golden age of X-ray and chemical crystallography. During his education his high school teacher got him interested in the sciences and World War II brought him to the group of the renowned crystallographer J. Monteath Robertson to do PhD research. He learned the art and science of structure determination: *art* because structures like the ones Jack worked on, with H, C and O atoms only, had first to be guessed at; direct methods had not yet been invented; *science* to the extent there were systematic heavy atom methods such as the Patterson method and the isomorphous replacement method, the latter invented by his supervisor. After gaining his PhD in 1944, his journeyman's years had him work with actual and future Nobel laureates, Dorothy Hodgkin in Oxford, Linus Pauling at Caltech and Sir Lawrence Bragg at the Royal Institution. He learned from the best. During his early career he published and joined ranks with the group of people who would constitute the core of chemical crystallography in the second half of the twentieth century.

In 1953 Jack married Barbara Steuer in Den Haag (Netherlands). The couple had two daughters, Marguerite (born 1955) and Julia (born 1957) and 68 happy years together. In their home in Küsnacht

(Switzerland) Jack and Barbara were superb hosts.

In the first half of the twentieth century, the developing field of X-ray crystallography started to provide basic structures at the atomic level for a broad variety of inorganic, organo–metallic, organic and biological compounds. These efforts together with progress in quantum chemistry – often at a qualitative level – culminated in new insights into the nature of the chemical bond as summarized in Pauling's (1960) book with the same title. Jack made lasting contributions to these efforts. On the basis of the semi-quantitative ligand field theory, Jack and Leslie Orgel explained structures of inorganic binary solids that were incompatible with the simple model of ionic bonding between charged spheres. Together Jack and Leslie also determined the unusual and surprising sandwich-type structure of ferrocene and characterized the metal–ligand bonding based largely on symmetry considerations. Ferrocene and its close relatives would remain a recurrent topic in Jack's Zürich research with P. Seiler. During an excursion into gas phase electron diffraction Jack and Verner Schomaker determined the structure of the cyclobutane molecule, among others. They confirmed the suspected elongated C—C bond distances and interpreted them in terms of repulsion between the non-bonded carbon atoms on the diagonal of the ring.

Around the middle of the twentieth century, the chemistry community, organic chemists in particular, realized that X-ray analysis had the potential for determining the constitution, configuration and conformation of their compounds. This would relieve them from the onerous task of structure determination by chemical degradation and free resources for improving their synthetic methodology. Jack's study with D. Hodgkin of a calciferol derivative settled its structure once and for all. At ETHZ, organic chemists and Nobel laureates L. Ruzicka and V. Prelog decided that X-ray crystal structure analysis, then primarily practiced in mineralogy, would be a fruitful physical chemical addition to their organic chemistry laboratory and Jack was to start such a group at ETHZ. Initially the 'infrastructure' of the laboratory consisted of a few X-ray generators, Weissenberg and precession cameras and a darkroom with boxes of photographic film. In the 1960s, this equipment was supplemented first by a linear and then a four-circle Hilger & Watts diffractometer, computer-controlled and equipped with an electronic point detector. These machines made visual estimation of diffraction intensities from films obsolete and reduced the time needed to measure diffraction data for a crystal – reflection by reflection – from months to about a week. The output of crystal structures increased dramatically, not least because in the early 1960s access to an ETHZ mainframe computer replaced the occasional night shifts at the computer of the local post office for solving structures with Patterson or direct methods and refining them with least-squares algorithms (at a rate of two or three cycles per night and often in block-diagonal approximation). With these technical advances it became possible to investigate families of related compounds in a reasonable amount of time, *e.g.* during a PhD project. Much more ambitious research problems could now be tackled routinely.

At the time of Jack's arrival at ETHZ there was no reliable quantum chemistry for quickly energy-optimizing structures of new molecules. Instead more or less successful interpolation schemes were invented. They took the form of empirical force fields whose structural and energetic parameters were optimized with experimental data. Their development required sampling bond distances, bond angles, torsion angles and non-bonded distances over as large a range as possible and combining structural with energetic information. At ETHZ, Jack started to determine structures of saturated, medium-sized carbon-ring compounds whose chemistry was actively studied in Prelog's laboratory. Connecting the ends of a chain to form a ring reduces molecular flexibility, some structural parameters become interdependent and are no longer free to assume their acyclic 'strain-free' values. Jack's studies of ring structures, many of them together with O. Ermer, were a rich source of information on strained bond and torsion angles. His results also answered tantalizing questions concerning unexpected trans-annular chemical reactivity and preferred arrangements of substituents. For the four-, five- and six-membered rings, Jack and J. Waser worked out the geometric constraints which take relatively simple algebraic forms.

The follow-up study on medium-ring lactams including some protonated forms was also based on the idea of ring constraints. The different ring sizes forced deformations of Pauling's planar peptide group, the detailed structure of which could be combined with other information to create an approximate potential energy surface of its non-planar deformations and *cis–trans* conformational interconversion. This work, performed with F. Winkler, may be regarded as an early example of 'structure correlation'.

The best known 'structure correlation' resulted from a collection of molecules showing a nucleophilic nitrogen atom at different distances from an electrophilic carbonyl carbon atom with an N···C=O angle of ~105°. Its discovery in 1972 was serendipitous. E. Shefter, an academic guest in the Dunitz group, collaborated with H.-B. Bürgi, then a young academic in the ETHZ laboratory of inorganic chemistry, on the crystal structure determination of methadone, at the time the main drug to treat heroin addiction. The backbone of the molecule was found to bend back onto itself to bring its amine nitrogen close to a carbonyl carbon atom. When Jack learned of the observation, he offered to scrutinize what he called 'the good old friends collection of literature data'. After a few days (without e-mail and without online access to the Cambridge Structural Database!) Jack came up with a handful of publications on compounds showing an N···C=O fragment in different stages of the approach of N to C=O. Together the molecules could be seen as mapping the reaction pathway of a nucleophile attaching itself to a carbonyl group to form a tetrahedral molecule. In the following years N···C=O interactions were invoked in a wide range of chemical phenomena, from stereospecificity of chemical reactions to stabilization of protein structures.

The concept of 'structure correlation' can be seen as a natural extension of Pauling's systematization of bond distances in terms of atomic radii and of the electronegativity differences between the bonded atoms. The concept is based on specifying a structural fragment, collecting all available structural data for the fragment in more or less different environments, and characterizing the distribution of its bond distances, angles and torsion angles in terms of a semi-quantitative energy surface. Such distributions usually show characteristic correlations among the different structural parameters, thus explaining the name of the concept. The principle was applied to tetrahedral molecules among others and led to conformational maps of conformationally flexible molecules. With the Cambridge Structural Database (CSD) a structure correlation study is now an almost trivial, undergraduate exercise. In the early 1970s, the CSD had a mere 2000 entries, but for Jack and his collaborators these were only accessible through the hardcopy book series *Molecular Structures and Dimensions*. In Zürich online access to the CSD had to wait until 1977.

Up to the mid-1990s most meetings on crystallography featured lectures or microsymposia on 'Structure Correlation'. An early overview of Jack's work in this field can be found in part two of his book *X-ray Analysis and the Structure of Organic Molecules* written in 1976, when Jack held the distinguished Baker Non-resident Lectureship at Cornell University (Dunitz, 1979, 1995). In 1994, Jack and H.-B. Bürgi edited a multi-author, two-volume book summarizing the topic (Bürgi & Dunitz, 1994). Talking about books, another one of Jack's publications must be mentioned here. For Jack, the crystallographer, symmetry was a *sine qua non* as it was for his chemistry friend E. Heilbronner. Together they wrote '*Reflections on Symmetry in Chemistry ... and elsewhere*' (Heilbronner & Dunitz, 1993), 'the best popular exposition ever written on symmetry and its role in chemistry' as R. Hoffmann wrote in his book review (Hoffmann, 1993).

The work on N···C=O interactions stimulated interest in looking at other non-bonded interactions, not just their geometry, but also their chemical context. Jack and his group, primarily W. B. Schweizer, M. Kaftory and J. D. Wallis, studied many related interactions between nucleophiles and electrophiles attached to the 1- and 8-positions of naphthalene. Nucleophiles and electrophiles were shown to have different directional preferences in their non-bonded contacts to divalent sulfur. These works are crucial forerunners of the still popular topics of halogen bonding, chalcogen bonding and all their relatives across the periodic table.

During the three decades following his retirement Jack started to question the meaningfulness of transferring the idea of atom–atom interactions, so successful in describing the (Pauling) chemical bond,

to non-bonded atom pairs. Do short intermolecular contacts necessarily imply stabilizing interactions? Together with A. Gavezzotti he ranked Desiraju's supramolecular synthons as stable, neutral or unstable, depending on the interaction energies between the fragment electron densities rather than between pairs of atoms in the synthons. From there it was but a small step to generalize the procedure to clusters of molecular charge densities and to estimate cohesive energies with contributions from their Coulomb, polarization, dispersion and Pauli repulsion energies. These ideas live on in a somewhat modified, extended form and are generally accessible in the widely used program *Crystal Explorer* with its numerical and graphical tools.

Jack liked problems, puzzles and mysteries. He did not always find the ultimate answer to open questions, but his essays on everything he wrote about are erudite, profound, interesting and entertaining.

One problem had to do with electron difference densities: as diffraction data became more accurate and differences between observed and model electron densities increasingly reliable, the question arose as to why some bonds in organic molecules showed an accumulation of (bonding) electron densities, while others did not. The problem lead to a flurry of activities by the charge density community and eventually to a more correct understanding of difference densities.

One puzzle Jack worked on with J. Bernstein: the disappearing polymorphs, *i.e.* crystal phases of substances that were routinely crystallized – some during long times – but unexpectedly vanished and only a different, usually more stable polymorph could be obtained and the original crystallization not reproduced. What seemed like a mystery initially could be broken down into pieces of a jigsaw. It was not entirely clear which pieces were essential and how to put them together, but the puzzle pointed to the necessity of searching for the unknown factors responsible for the lack of reproducibility. Controlling polymorphism has become an important aspect of drug development. Crystallization, especially the events during nucleation, are still largely uncharted territory.

One mystery, one that remains largely mysterious, is that in catalogs of organic chemicals, molecules with an even number of carbon atoms are systematically more numerous than those with an odd number. Why?

Jack pursued many other scientific interests such as the ion-selectivity of ionophores (with M. Dobler). The study with R. Taylor entitled 'Organic fluorine hardly ever accepts hydrogen bonds' is Jack's most cited paper. With K. Trueblood, E. Maverick and C. P. Brock, all three regular sabbatical guests in the Dunitz group, he studied atomic and molecular motion in crystals. With his last PhD student, E. Nowotny-Bregger, Jack sought to elucidate the molecular motion required for a first-order phase transition. Although much experimental information was accumulated, Jack concluded: 'I have no explanation of these remarkable results'. Some 25 years into his retirement Jack hoped to breathe new life into the topic: 'I had hoped to find on the Internet an up-to-date review of what is known today about first-order phase transitions in organic crystals. My search was a disappointment. This branch of science, which still seems so interesting to me, does not appear to be a very popular area of study. ... I write this little essay, however, with the hope that it may stimulate some young scientist to reexamine these problems with modern methods and especially with new ideas and thereby to fill some of the gaps in our understanding' (Dunitz, 2016).

During his time at ETHZ Jack was part of a tightly knit group within the laboratory of organic chemistry. He partnered, among others, with A. Eschenmoser for studies of corrins and enamines and with D. Seebach for studies of Lienolates and other organic Li salts. Their friendship in science and in life helped shape an institution that was far more than the sum of its parts. Many world-renowned academic guests, not just crystallographers, and many postdocs visited with Jack's group or with the laboratory of organic chemistry. When they joined the lunch table of the Dunitz group all sorts of topical, challenging and seemingly crazy questions were discussed. For example, who would want to know how many locomotives the Swiss railroads operate? Probably nobody, but the discussion taught the lunch guests how to make reasonable guesses, individually or as a group, about problems of which they knew next to nothing. This

author benefited from the spirit in the Dunitz group and the ETHZ laboratory of organic chemistry during four years in Jack's group as a PhD student and, after returning from a postdoctoral stay, during another eight fruitful years as a young academic. An important part of the collaboration with Jack was paper writing in his office during lunch time. In some sessions no more than a single sentence was discussed, eventually written down and often modified the next day. Clear language was important to Jack. His preoccupation with texts goes with his interest in literature. His attitude lives on in Dunitz's rule: 'Almost every scientific paper can be improved by deleting the first sentence.'

Jack's contributions to chemical crystallography were widely recognized: he was awarded the centenary Prize of the Royal Society of Chemistry (1977, with G. Olah and K. S. Pitzer), the Paracelsus Prize of the Swiss Chemical Society (1986), the Gregori Aminoff Prize of the Royal Swedish Academy of Sciences (1990), the Buerger Award of the American Crystallographic Association (1991), the Havinga (1980) and Bijvoet (1989) medals. Jack was elected Fellow of the Royal Society of London and member of the German Academy of Sciences Leopoldina, the Academia Europaea and the European Academy of Sciences and Arts; he was a foreign member of the Royal Netherlands Academy of Sciences, the US National Academy of Sciences, the American Philosophical Society, and the American Academy of Arts and Sciences. Jack was awarded honorary doctorates from Technion (1990), Weizmann Institute of Science (1992) and the University of Glasgow (1999), and honorary membership of the Swiss Society of Crystallography, the Royal Society of Chemistry, the Swiss Chemical Society and the British Crystallographic Association.

Jack expressed his stance towards crystallography in a short essay on the future of crystallography: ' ... if crystallography has an uncertain future it has a great past and it still possesses the rare intellectual charm of relating to the three basic sciences, physics, chemistry and mathematics, as well as to the decorative arts. It should not be taught or studied in a too restrictive, introspective manner' (Dunitz, 2002). Jack's writings express this 'rare intellectual charm' and he and his work certainly are an integral part of crystallography's 'great past'.

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1. Part 1: https://www.youtube.com/watch?v=bCTMoUEH5ts.

1. Part 2: https://www.youtube.com/watch?v=jN03OQLfjrI

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For a list of papers by Professor Dunitz appearing in IUCr journals click here.

OBITUARY

Kenneth C. Holmes (1934–2021)



John Wray, Ilme Schlichting

Ken Holmes was born in England and studied physics at St John's College, Cambridge, from where he graduated in 1955. His initial interest in crystallography led him to apply to the Department of J. D. Bernal at Birkbeck College, London (part of London University). Bernal was a pioneer of research on the molecular structure of living material, which triggered Ken's specialization in diffraction by fibrous materials.

In 1955 he started his PhD under Rosalind Franklin. His thesis work focused on the tobacco mosaic virus (TMV), for which Bernal and Fankuchen had, as early as 1936, found clear evidence of remarkable regularity using X-ray diffraction. This regularity was known to be that of a helix; as Ken noted many years later, this was not quite what he had understood by crystallography! His work focused on the equator of the pattern and, in a first use of isomorphous replacement with a fibrous structure, obtained an image of the projection down the helix axis. Aaron Klug had already joined Franklin's group in 1953, and Don Caspar soon became a regular visitor there also. Aaron and Don became life-long friends for Ken, and Don died last year, only a few weeks after Ken.

Franklin died in 1958, and Ken finished his PhD under Klug's leadership in 1959. He and his wife, Mary, then spent 15 months in Boston, MA, USA. Ken had planned to work there with Don Caspar at the Children's Hospital, but his main project ended up being with Don's colleague Carolyn Cohen on the structure of paramyosin in the ABRM: Ken's first introduction to muscle. Ken and Mary returned to England in time for the birth of their first child. Ken received an appointment at the relatively new MRC lab in Cambridge, where Hugh Huxley had for some years been studying the structure of muscle, and the central goal was soon to obtain diffraction patterns from living muscle fibres. This work focused on the construction of X-ray sets with rotating anodes to allow greater intensity. In 1963 Mike Reedy arrived as a postdoc, and he and Ken, together with Richard Tregear, started an investigation of insect flight muscle: intact but permeablized. This preparation was ideally suited for studying structure (by X-ray and EM), mechanics and biochemistry and led to a major breakthrough: the finding that the appearance of the 'crossbridges' depended on the absence or presence of ATP, the energy source for contraction. By the mid-1960s, Ken was looking for a permanent position, and he was considered for a directorship at the Max Planck Institute for Medical Research in Heidelberg, Germany. He had had contact with the MPI for Virology in Tübingen, and the idea of Heidelberg was attractive. He knew, moreover, that one of the world's first synchrotrons was in Hamburg.

Ken was duly appointed and moved to Heidelberg in September 1968. His projects there continued to concern diffraction from fibres, namely muscle filaments and the TMV particle. The crystallography of TMV was progressing in Cambridge: disk-like aggregates of the protein subunit had been found, allowing the structure of the subunit to be solved crystallographically. The structure of the native helix was

pursued (with Stubbs and Warren) using further derivatives, leading finally to a refined structure of the native rod-shaped particle. The work of Ken's department progressed in several other directions: developing more intense X-rays for observing changes in structure during contraction, the structure of the actin and myosin filaments of striated muscle, and the structural analysis of other proteins which, like myosin, split nucleotides.

Ken's move to Heidelberg immediately opened a window of opportunity regarding intense X-ray sources. His student Gerd Rosenbaum had studied at DESY in Hamburg and knew the situation there − and support was available from the DESY directors, who sensed that the prospect of biological applications would raise further support for DESY. (A few years later, the situation reversed: Ken's initiative at the DESY outstation made Heidelberg an attractive site for the newly founded EMBL.) Ken and Gerd spent long periods at DESY and finally established the world's first beamline there. Beamlines at synchrotrons world-wide followed from this work, and the resultant advances in crystallography have since then revolutionized structural biology.

The second challenge was the crystallography of actin. The native actin filament resembled TMV in being a non-integral helix, but was variable and gave fibre diffraction patterns with too little resolution for determination of the atomic structure. Its globular monomer moreover resolutely refused to form crystals, but did so as a complex with DNase. The analysis of this involved great practical difficulties, but the structures of the actin monomer and filament were finally obtained in 1990.



Work in Ken's department had involved EM approaches to the structure of myosin also, and ultrastructural studies of nucleotide analogs had raised tantalizing glimpses of a possible structural basis of the force-producing event in muscle. It was a minor sensation when, in 1993, a first X-ray crystallographic structure of subfragment 1 of myosin, the 'head' of the molecule and the 'crossbridge' in muscle, became available from others. The challenge now was less in crystallography than in the interpretation of the results, and Ken was crucially involved in the fitting of the structure into EM images of actin filaments with myosin attached. Within a few years, several groups had presented clear evidence

for polymorphism of the head structure, and intense discussions with several groups converged on a consensus of how myosin and its essential partners (ATP, its hydrolysis products, and actin) produce force and movement. Ken provided a crucial insight that the original S1 structure had been incompletely interpreted and, in reality, presaged the essential changes in myosin's conformation − and thus the mechanism of force-generation in muscle.

Ken retained the physicist's eye for essential questions and crucial simplifications. He enjoyed ruthlessly ignoring details and was always ready to abandon well-trodden paths when (or even before) the need arose. He died on 2 November 2021. His energy, enthusiasm, clarity and inventiveness will be greatly missed.

John Wray and Ilme Schlichting are at the Max Planck Institute for Medical Research, Heidelberg, Germany.

Further reading

Holmes, K. C. & Rosenbaum, G. (1998). How X-ray Diffraction with Synchrotron Radiation Got Started. *J. Synchrotron Rad.* **5**, 147–153.

OBITUARY

Vasyl Kladko (1957–2022)



Present and former collaborators from V.E. Lashkaryov Institute of Semiconductor Physics (Kyiv, Ukraine) and from the Institute of Physics PAS and Institute for High Pressure Physics PAS (Warsaw, Poland)

We announce, with deep sadness, that on 13 March 2022 Russian aggressors killed the experimental physicist Professor Vasyl Petrovich Kladko (65), a famous Ukrainian crystallographer specializing in high-resolution X-ray diffraction and its application in studies of low-dimensional systems. He was a victim of an attack by Russian soldiers on noncombatants while being evacuated from the besieged Vorzel/Irpin settlement near Kyiv, Ukraine. Fortunately, at the moment of the attack, his wife and grandchildren safely escaped from their house, where they (like hundreds of other Irpen citizens) were hidden for a number of days under awful conditions in dark cellars without food, water or electricity.



Professor Vasyl Petrovich Kladko.

Vasyl Kladko was born on 12 January 1957 in the village of Ozero in the Rivne district, Ukraine. He earned his Masters Degree in Chernivtsi University (Chernivtsi, Ukraine) in 1979 and completed his PhD in 1986 and DSc in 2000, both in the V.E. Lashkaryov Institute of Semiconductor Physics, National Academy of Sciences of Ukraine. In 2007 he was awarded the title of professor. In 2015, Vasyl Kladko was elected a corresponding member of the National Academy of Sciences of Ukraine.

For a long time, Vasyl Kladko served as head of the department and as deputy director of the V.E. Lashkaryov Institute of Semiconductor Physics. He was awarded a number of prizes, in particular from the State Prize of Ukraine in the field of science and technology.

For many years, Vasyl Kladko worked on high-resolution X-ray diffraction, and made valuable contributions throughout his long career in applying these methods to analysis and diagnosis of low-dimensional systems, particularly defect structure, morphology and relaxation of thin films and multilayers. During recent years he also participated in studies of materials suitable for infrared detectors. He promptly collaborated with scientists from Poland, the Russian Federation and others – this is reflected in his publications. His work led to more than 300 papers coauthored by him as well as some monographs, in particular *X-ray dynamical diffraction in multilayers* by O. Yefanov, V. Kladko, V. F. Machulin and V. Molodkin (in Ukrainian; Naukova Dumka, Kyiv 2008).



Professor Vasyl Kladko (on the far right) with six members of his X-ray diffraction group (during happier times in December 2021).

We shall all sorely miss him. With his death, Ukrainian and world science is experiencing a huge unjustified loss. Our thoughts go to his wife Galyna and his whole family.

IUCR SUPPORTED EVENTS

IUCr supported meetings

These forthcoming meetings are supported by grants from the IUCr Calendar Committee.



11th Oct 2026 - 16th Oct 2026

Indaba10

Skukuza, Kruger National Park, South Africa



5th Dec 2026 - 10th Dec 2026

African Materials Research Society (AMRS 2026)

Nairobi, Kenya

MEETINGS CALENDAR

Meetings calendar

This is a list of meetings of interest to crystallographers occurring within the next twelve months, as recorded in the IUCr Calendar of Events. Please let us know of any that are missing by completing this form or sending an email to forthcoming.meetings@iucr.org.



Indaba10 South Africa 11th Oct 2026 - 16th Oct 2026



Synchrotron Radiation: Science, Knowledge, Innovations, Future (SKIF-2026) Russian Federation 18th Oct 2026 - 23rd Oct 2026



CCP-EM/Diamond Icknield Workshop United Kingdom 9th Nov 2026 - 13th Nov 2026



Instruct-ERIC hands-on workshop on Advanced cryo-FIB-SEM methods Germany 9th Nov 2026 - 13th Nov 2026



Crystal36-AXAA 2026 Meeting Australia 26th Nov 2026 - 2nd Dec 2026

African Materials Research Society (AMRS 2026) Kenya 5th Dec 2026 - 10th Dec 2026

Joint Polish-German Crystallographic Meeting 2027 Germany 15th Feb 2027 - 18th Feb 2027